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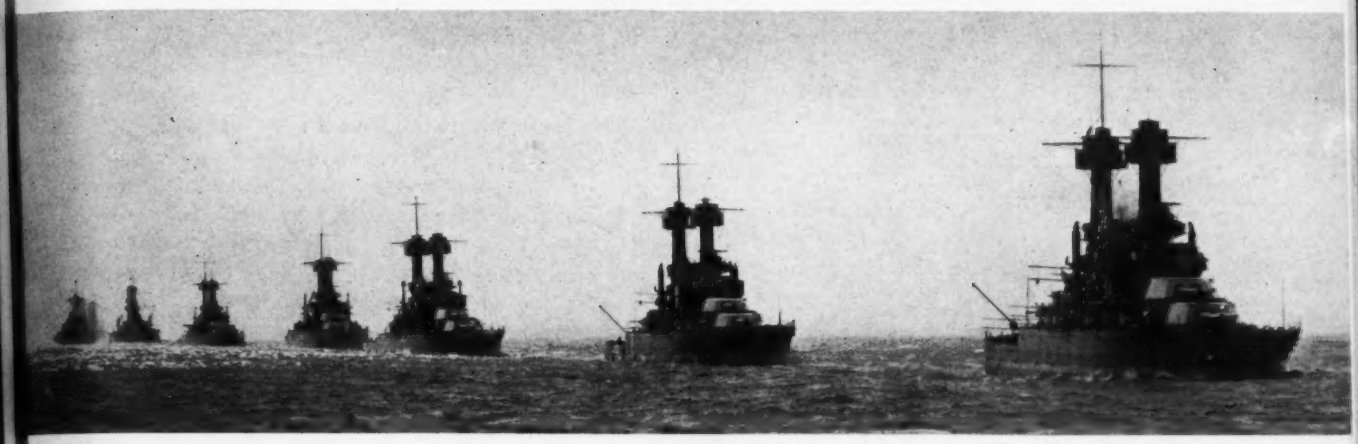

Textiles in the Navy

By **FREDERICK KRASSNER**
Chief Chemist, Naval Clothing Depot, Brooklyn, N. Y.

Among the fundamental needs that must be adequately provided for in the Navy are such primary requisites as food, shelter and clothing. The Navy is, of course, primarily an organization of men and ships, the first line of defense in time of war, and in time of peace an insurance policy, as it were, against war. In order to maintain such an organization at a high point of efficiency, the foregoing needs must be taken care of at all times, both ashore and afloat. It will be the purpose of this article to discuss some of the aspects of not the least important of these needs, viz: the problem of clothing, the enlisted personnel of the Navy, and the procurement of such textile items as are needed for their comfort and well being. Some of the scientific methods used in evaluating the foregoing, will be outlined, as well as the general contributions that are being made to science and industry in this field.

IT is well known that the Navy Department has always had "one of the most complete and best articulated set of supply standards and specifications of any organized body either governmental or private."* Similarly as regards its system of inspection of various deliveries, a rigorous supervision of the quality of all such deliveries is maintained, including the inspection of the various textile items procured. In the Navy Department, it is realized that any group of materials worthy of a complete set of specifications is also worthy of intelligent inspection by a competent inspection staff. In the long run, it has been found, that such procedure results in greater economies insofar as inferior materials are not accepted. Moreover, rigid inspection meets with the hearty approval of reputable manufacturers who are thus assured that they are not competing in price with inferior quality.

In its relation to the world of business the Navy stands mainly as a buyer or consumer, with purchases in the textile field running into millions of dollars a year, in order to meet the needs of the Navy. Not only does the foregoing include the various items of the enlisted man's wearing apparel such as blue uniform cloth, white drill, chambray, denim, finished articles such as socks, underwear, neckerchiefs, hats, shoes, raincoats, etc.; but also such items as blankets, sheeting, mattresses, pillows, tablecloths, towels, canvas materials, furniture covers, bunting and other similar articles too numerous to mention. The Bureau of Supplies and Accounts which is the central purchasing department of the Navy, endeavors to coordinate its annual purchases of textiles to market conditions, which results not only to the Navy's advantage, but also for the good of the industry as a whole.

How does the Navy Department insure the quality of all the textile materials procured? Modern testing devices have eliminated to a great extent any element of guess work in the purchase of such materials, all deliveries of which are tested scientifically for their intrinsic value. An up-to-date Laboratory, modernly equipped with such scientific instruments

* National Industrial Conference Board, Industrial Standardization, 1929, p. 130.

as are needed to insure conformity with purchase requirements, maintains a careful scrutiny of all articles delivered. Such characteristics as the following which are determinable without reference to a specific use to which the material may be subjected—are definitely measured,—tensile strength, weight, color, size, identification of dye, and composition. The character of the yarn is measured and described in terms of the number of ply, number of twists per inch, and count or size of the yarn used. The general qualities of construction, finish and workmanship can be evaluated with a fair degree of accuracy.

In the past most textile testing was usually limited to such tests as enumerated above, which afford a general criterion of the quality of textiles for certain purposes. Today, however, the technical department is more interested in the practical question of how the material will stand up under practical service conditions, which in the Navy are somewhat more rigorous than ordinary commercial usage. In this category fall such characteristics as the following: shrinkage, durability, color measurement, fastness of dye, inflammability, waterproofness, coefficient of friction, accelerated ageing, ability to withstand laundering and dry cleaning, etc. The measurement of the foregoing performance characteristics necessitates reproducing artificially the approximate conditions to which such materials may be subjected in practical usage. Evaluation of such characteristics, in the final analysis, affords valuable information as to what serviceability may be expected from a given textile, whether it be in the fabric of an enlisted man's wearing apparel, in the blanket used to cover him at night, or in some form of aeronautical material upon which human safety may depend.

While the enlisted man's uniform as such, has never failed to attract the attention of the public eye, conveying perhaps romance of adventure into wide ocean spaces and far off lands, it can be seen that the procurement of the different articles constituting it presents certain practical considerations. It should be pointed out that if an article issued to the fleet proves unsatisfactory in its serviceability, whether on board ship or in some distant station, it is usually returned and placed under "survey," which results in disposal at a great loss. The rigid system of inspection, previously referred to, serves therefore not only in checking conformity with specified requirements, but aids in reducing such losses to a minimum. Let us examine a few other typical illustrations of tests performed, and some of the problems arising in connection with them.

Strength

Strength of fabrics is determined by means of a standard tensile strength testing machine, which measures the strength of the fabric in terms of the number of pounds required to stretch the threads to the breaking point, furnishing information at the same time as to the stretchiness and elasticity of the material. As a prerequisite to this test, especially in the event of a controversy, standard atmospheric conditions are maintained in the test room, the latter being essential for dependable and speedy results. All similar physical tests on textiles are conducted in this special conditioning room, which is maintained at a constant temperature of 70° F. and a relative humidity of 65 per cent. The system for maintaining these constant conditions is of the latest type, and is entirely automatically controlled.

Fastness

In the case of dyed materials, fastness tests constitute an important group of tests, after the dye has been properly identified by means of chemical analysis. How fast is the dye to sunlight, to laundering, to perspiration, to crocking, etc.? These are some of the factors that must be evaluated separately. Fastness of the dye to light is ascertained by exposure to artificial sunlight in the standard sunlight machine, viz: the Fade-Ometer. Fastness to a washing is determined in a Launder-Ometer, which is the equivalent of 20 washing machines in 1 unit. In this test the effects of soap, water and high temperature are shown, which are judged by the amount of bleeding onto white material and into the soap solution, as well as any loss of color on the fabric itself. Changes of color are compared with the original color in a Coloroscope, which measures color with the aid of the photo-electric cell. This machine is independent of daylight and individual eyesight, indicating automatically the difference in intensities between two samples. As a basis of judging comparisons between deliveries and standard sample, the instrument furnishes valuable information. Uniformity of color has always been a problem in military organizations, where an opportunity is afforded to make close comparisons when men are lined up for inspection, and where slight deviations in shade are more apparent than elsewhere. It is realized that considerable painstaking research, and the accumulation of accurate data on a large variety of colors is essential, before accurate standards and exact tolerances, can be set up, correlating expert visual comparison with scientific measurements. Investigations along these lines are under way, considerable progress having been made to date. Navy blue cloth, which is extensively used in the manufacture of Naval uniforms, is one of the major colors under consideration.

Navy Blue

It may be of interest to divert somewhat at this point and present a brief resumé of navy blue as a color in the Navy. Due to its depth of tone and intensity of shade, which afford great durability, navy blue has always been regarded as a very practical color. There exist, however, a variety of navy blues, although, in general, it may be said that the standard navy blue is a very dark shade of blue possessing a violet tone. This is the standard adopted by the Navy Department and is based upon a combination of two dyes, which have been found necessary to furnish the desired tone. Formerly indigo alone

was rather extensively employed for the dyeing of Navy Blue; but it was found to be insufficiently violet in tone by itself. It was therefore necessary to have it combined with some suitable dyestuff that was properly fast and which would at the same time provide the necessary correction to the tone of the color. For this purpose dyers used alizarine blue, which has been found to fill the bill very satisfactorily. Navy specifications, therefore, call for a combination of Indigo and Alizarine as the dyes to be used on the enlisted man's uniform cloth.

Perspiration

Fastness of dye to perspiration is another factor that must be evaluated especially in connection with socks, which are dyed black. The effect of acid or alkaline perspiration on the dye is determined by contact, under prescribed conditions, with solutions representing average perspiration conditions. Fastness to perspiration is judged by the amount of staining of white cotton and wool. Up to a few years ago sulfur black was specified as the dye for socks. One of the difficulties arising from the use of sulfur dye was the tendency after storage for some time of the fibers to become tendered, and the socks unsuitable for issue. This tendering was the result of a gradual development of Sulfuric Acid by the oxidation of the sulfur and was responsible for considerable losses. Investigations were therefore conducted as to a more suitable dye that would not only meet the fastness requirements, but also resist deterioration. Developed blacks were found to meet the problem satisfactorily and have since been specified on all blacks.

Shrinkage

The problem of shrinkage encountered in various textiles is an important one; both from the standpoint of yardage procured and wearability of the finished garment. In an effort to minimize laundry shrinkage of fabrics as much as possible, a study of commercial methods of preshrinkage was initiated some time ago. Inasmuch as the causes of the shrinkage of fabrics when subjected to a full laundry treatment are mostly mechanical, it was concluded that the most effective treatment to prevent shrinkage should be found in a process involving the mechanical rearrangement of the fibers of the fabric to the same extent that the fibers would arrange themselves if subjected to a full washing in the laundry. Accordingly, the principle embodied in the Sanforizing process was considered satisfactory for preshrinking such materials as Navy drill, chambray, nainsook, denim, etc., all of which enter into the enlisted man's wearing apparel. All such items are now procured on a pre-shrunk basis, assurance being thus made in advance that there will be no further shrinkage in actual use. Materials procured on this basis are subjected to a rigid shrinkage test, which simulates actual laundry procedure, deviations in shrinkage in excess of the tolerance allowed being a cause for rejections. The shrinkage test used is the result of extensive tests conducted by the Navy and in modified form is now an official test in all Federal purchases. Investigations are at present under way in connection with woolen goods, and it is anticipated that in the near future the wool uniform cloth will also be procured on a pre-shrinkage basis by the Navy Department.

Blankets

As regards the procurement of blankets, warmth and durability are the main characteristics desired. The physical properties of a blanket are influenced by the kind of fiber used in making it, as well as the general construction. Only all-wool blankets are used in the Navy, and they must be free from shoddy, waste, vegetable matter and other impurities. Durability of the blanket is an important factor in its construction. In general, the more nap the blanket has the warmer it is, although excessive napping weakens the blanket, the yarn tending to pull apart when subjected to strain. The blanket must therefore possess the right amount of napping consistent with the requirements. An examination of the enlisted man's blanket will reveal that it is unlike ordinary commercial blankets in appearance, not having the usual amount of nap encountered in the latter. However, in a recent comparative survey of blankets sold in a large metropolitan department store, the Navy blanket showed the highest relative warmth, as revealed scientifically by an instrument measuring such warmth in terms of thermal transmission. Of interest also in this connection is the fact that the Navy blanket was being procured at a relatively lower cost. Navy sheeting, similarly, is purchased under definite specifications as to thread count, strength and weight. No added sizing is permitted, which might make the sheeting appear heavy and give it a smooth finish; but which after laundering would become sleazy and fuzzy in appearance. Other items of bedding include pillows and mattresses, in connection with which cleanliness and quality are inspected.

Aeronautical Fabrics

In the field of aeronautical fabrics, which are under the cognizance of a separate department, a number of serviceability requirements must be met. It is of great importance that such materials as parachute cloth, the cloth used for the outer cover and gas cells of dirigibles, and airplane wing cover cloth, have a minimum weight for the required strength, and also offer resistance to tear. Air permeability is a vital characteristic of parachute cloth and must be held within a fairly definite and narrow range. In measuring the amount of air passing through a given area of cloth, a scientific apparatus is used, which measures the foregoing at a definite pressure, within a specified time. It can thus be ascertained in advance whether the cloth is likely to rupture due to inability to resist air pressure when the cloth is too impermeable, or on the other hand, whether due to excessive permeability the descent may be too fast for comfort and safety. It is interesting to note that practical service tests conducted by the Navy Department of parachutes made from special

cotton cloths, have shown them to be reliable and serviceable so that in the event of an emergency curtailing the supply of silk, all of which is imported, parachutes could be and are being made for commercial purposes from the domestic fibers.

Standardization

It is not within the scope of this article to cover all the items purchased and describe the various tests enumerated. In general, standard official tests are used, the technical department being constantly at work devising new tests and special tests for specific purposes. The increasing interest in consumer standards, together with the modern use of time and performance guarantees in merchandising, has resulted in research and control along the lines of standardized and scientific methods of testing. It should be borne in mind that although considerable progress has already been made in the field of textiles, standardization in this field is still in the developmental stage. The technical department of the Navy is not only keeping pace with the developments, but it is pioneering in certain directions. Today, all over the country, industrial and consumer research bureaus are springing up, prepared as the Navy Department has always been, to evaluate serviceability in textiles.

● CIBA CIRCULARS

The Society of Chemical Industry in Basle, represented by the Ciba Co., Inc., has lately issued the following circulars describing new products, which may be obtained from any of the offices of the company:

Circular No. 411 *describes Sapamine KW Pat.* This is said to be a distinct novelty in auxiliary products as it fastens acid and direct dyeings to water, even boiling hot and acid water, permitting cross-dyeing with ordinary direct dyes. It is said to exhaust from the bath and impart a silky hand to all the common textile fibers, producing a finish that, it is claimed, cannot be duplicated with soap and oil mixtures. It is clean and odorless and may be applied either in the rinse bath or in the finishing room. It is said to be of great value in the continuous washer in the print department as its soaping nature assists in washing out the gum, the ground is fastened and application prints are fixed, while the fabric is softened. It further aids in the level dyeing of basic dyes, whether on a mordant or in topping direct and sulfur dyes.

Circular No. 415 *describes Chlorantine Fast Blue 2GLL.* This addition to the series of Chlorantines of highest fastness to light is clearer and redder than the 3GLL type. It is said to dye cotton and rayon, but reserve all acetates, even in large patterns. Silk is also said to be reserved in a slightly alkaline bath and wool nearly so. The dyeings are dischargeable to a pure white with hydrosulfite.

Circulars Nos. 416 *and* 417 *describe Albatex BD.* The principal use of this product is for addition to the alkali in kier boiling, whereby vat dyes are said to be prevented from bleeding into the white. Albatex BD is also used as a reserve in printing vat dyes and discharging as it is said to neutralize the effect of reducing materials.

Sample card No. 1148 *is titled Neolan Dyestuffs on Hair Hats.* This card is illustrated by 24 shades, made in practical dyeings, of seasonable colors on hair felt, which have been dyed with Neolan dyestuffs. The ability to dye the fast Neolan dyestuffs on fulled hat bodies is said to be regarded as a great step forward in the hat industry and should prove to be of great interest in this country.

● AWARDED A. I. C. MEDAL

The medal of the American Institute of Chemists, presented annually for outstanding service to chemistry in America, was awarded this year to Dr. James Bryant Conant, President of Harvard University, according to an announcement made recently by President Henry G. Knight from the Institute's office at 233 Broadway. The award is made in recognition of Dr. Conant's many contributions to chemical science.

Dr. Conant has done notable work in establishing the chemical structure of many complicated organic compounds, including among others, haemoglobin of the blood substance, chlorophyll—the green coloring matter found in plant life, and a number of other coloring substances occurring in flowers and feathers.

Last year's medalist of the American Institute of Chemists was Dr. Henry C. Sherman, Head of the Department of Chemistry at Columbia University, whose outstanding researches in the fields of food chemistry and vitamins are well known. Other past medalists include Dr. Charles H. Herty, internationally known paper chemist; Andrew W. Mellon and Richard B. Mellon, founders of the Mellon Institute for industrial research; the late George Eastman, who made fine organic chemicals available to the chemists of this country; and Mr. and Mrs. Francis P. Garvin, who established the Chemical Foundation.

The medal will be presented to Dr. Conant at the annual meeting of the American Institute of Chemists, which will be held at the Chemists Club in New York today, (May 21st). Dr. Hans T. Clarke, F.A.I.C., Professor of Biological Chemistry at Columbia University, a friend and associate of Dr. Conant for many years, has been selected to make the presentation. Dr. John H. Finley of the *New York Times* will be among the speakers of the evening.

● COLOR

An interesting study in color is shown at the Bride's House, recently opened to the public at 444 Madison Ave., New York City, by House Beautiful combined with Home & Field.

Persons interested in the study of color and color schemes will find the rooms of interest. The exhibit will be open for three months.

Recent Developments in the Chemistry of Silk and of Silk Processing--III

(Continued from page 254, May 7th issue)

By **WALTER M. SCOTT, Ph.D.**
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CHAPTER III

THE DEGUMMING OF SILK

THIS is the process in which the outer coating of sericin (silk gum) is removed from the fibroin (silk fiber). However, the removal of sericin is not the only thing which should be accomplished in this degumming operation. The pure silk must also be freed from the natural fats and oils left by the silkworm, as well as from the fats and oils added by the throwster. And finally, the natural pigments, as well as the throwster's tints, must be eliminated as completely as possible. All of these factors should be kept in mind when comparing the various methods recommended for degumming.

Degumming with Soap Solutions

By far the greater proportion of processed silk has been, and still is degummed in a boiling or nearly boiling solution of neutral soap. The proportion of soap is usually about 15% on the weight of the silk, or from 1 to 2% on the weight of the water. It has been determined that the following factors play a part in the degumming of silk by means of soap.

1. *The pH of the soap solution.* It has lately come to be realized that this is one of the most important factors in this operation. According to Mosher the most satisfactory results are obtained when the pH is between 10.0 and 10.5. Below 10.0 the action of the soap on the sericin slows up very rapidly, and above 10.5 the tensile strength of the fibroin is diminished appreciably.

2. *Temperature.* Very little degumming takes place below 180° F., but the action increases rapidly above this temperature. The maximum speed of removal of the silk gum is between 200 and 212° F.

3. *Time.* The time required to complete the degumming operation, naturally varies with the changes in the other factors, and with differences in the nature of the raw silk. In general, treatment for one to two hours in a boiling soap solution whose pH is about 10.2, should be sufficient. There is no gain in the quality of the finished product by lowering the temperature of the bath and increasing the time, or by raising the alkalinity of the bath and decreasing the time.

4. *Type of soap.* Soaps made from olive oil, or from red oil (commercial oleic acid), are most commonly used for the degumming of silk. Soaps from oils of low titer are preferable because they are easier to dissolve and may be rinsed out of the silk more readily. Moreover, small amounts of lime soaps from low titer oils are easier to get rid of without damage to the silk. Recent experiments by a Japanese investigator show that soaps from unsaturated fatty acids (such as oleic acid) leave the finished silk more lustrous than soaps from saturated fatty acids (such as stearic and palmitic acids). The saturated fatty acid soaps are slightly more efficient in removing the sericin because of their higher percentage of hydrolysis, but this is of very little significance in commercial practice because there is always an excess of soap present in the degumming bath.

Mechanism of the Soap Degumming

Both free alkali and free fatty acids are present in the degumming bath due to hydrolysis of the soap solution. Fibroin and sericin are amphoteric i.e. they have both acidic and basic properties. Hence they are capable of combining with both the alkali and the fatty acids of the soap. However, in actual practice much more alkali than fatty acids is removed from the soap solution and the pH is always reduced during the degumming operation, Sericin is much more sensitive to alkali than fibroin, and it is this property which enables the sericin to be removed without serious injury to the fibroin.

A recent paper reports that at least 1% of soap or its hydrolytic products remains combined with the silk fiber, either physically or chemically or both, and influences the nature of the finished silk. Alkali destroys the luster of the silk and decreases its tensile strength and elongation; while fatty acids increase its elasticity and are the underlying cause of scroop.

With a proper understanding of the nature of the action between the raw silk and the soap in the degumming bath, it is possible to make the same soap solution do for the removal of sericin from several lots of silk. In 1925 the writer reported that it was possible, after

one lot of silk had been degummed, to add sufficient caustic soda to the boil-off liquor to bring it back to its initial pH, whereupon another lot of silk could be satisfactorily degummed in the same solution. It is now quite common practice to degum several lots of silk in the same soap solution, by bringing the pH back to about 10.2 before the degumming operation, and adding a small amount of fresh soap, if necessary, to replace that which had been taken up by the silk.

Other Alkaline Degumming Agents

Potash soaps have been recommended from time to time as substitutes for soda soaps. They have the advantage of easy solubility and are said to produce more lustrous silk than soda soaps. However, in the experience of the writer, the advantages to be gained by the use of potash soaps in the degumming bath were doubtful, and in any event were not sufficient to justify their increased cost.

A recent patent claims that sericin may be dissolved in a solution of monoethanolamine soap in the presence of an excess of the amine. Soaps of triethanolamine have also been mentioned in this connection, but have not been extensively used. In the case of soaps of mild alkalies such as these, it is necessary to add some other agent in order to raise the pH of the solution to the most efficient degumming range.

It is possible to dissolve sericin in solutions of any of the common alkalies such as: sodium hydroxide, sodium carbonate, sodium silicate or trisodium phosphate, provided that they are present in sufficient concentration to make the pH of the solutions 10.0 or over. However, such alkalies by themselves are not satisfactory for the degumming of silk for the following reasons: (1) they tend to attack the fibroin also and cause a marked decrease in the luster of the degummed silk, (2) they do not remove the natural fats and waxes as well as soap solutions, and (3) they leave the silk limp and do not give it the "body" which is provided by the absorption of a small amount of soap. A recent paper reports that there is a marked decrease in the viscosity of fibroin after it has been boiled off in a solution of sodium carbonate, but very little change in viscosity after boiling off in soap solutions.

A novel suggestion was made by the I.G. in a recent German patent. They propose to degum silk by treatment with ammoniacal solutions of zinc hydroxide or cadmium hydroxide. However, it would seem as if there might be some difficulty in confining the action of this solution to sericin alone.

Boil Off Oils

In the chapter devoted to the soaking of silk mention was made of the development of sulfonated oils as well as other emulsifying and penetrating agents. Coincident with the application of these compounds to the soaking of silk, studies were also made of their possibilities in connection with the degumming of silk. These researches led to the preparation and marketing of the so-called "boil off oils."

Most of these oils depend upon some form of alkali for the principal degumming action, but modify the alkali with a sulfonated oil. The function of the oil is to tone down the harshness of the alkali, assist in the removal of the natural fats and waxes and furnish the necessary fatty matter for absorption by the fibroin. In general, either sulfonated castor oil or sulfonated oleic acid is used in the boil off oils, although mineral oil sulfonates have also been recommended in several recent patents.

Certain organic solvents as well as cresylic acid, have been used in combination with sulfonated oils in certain boil off oils.

It is easy to visualize the possibilities of the sulfonated fatty esters or the fatty alcohol sulfates in connection with the degumming of silk, but much research work must be done before their advantages and disadvantages are fully known.

Degumming in Acid Solutions

It is possible to remove a large proportion of the sericin from silk in an acid solution. According to Mosher the optimum pH range for degumming silk on the acid side is between 1.75 and 2.5. Unfortunately acids do not remove the natural fats and waxes, nor the natural coloring matter of the silk.

It is difficult to see any advantage for acids over soap in the degumming of all silk fibers or fabrics. However, it has been pointed out that in the case of silk-wool unions, the usual boiling soap treatment is somewhat detrimental to the wool. For mixed fabrics of this type it is suggested that a preliminary acid degumming followed by a wash and a mild soaping, might result in a finished product of higher quality. Further studies are required to settle this point.

Degumming with Enzymes

It has long been know that certain enzymes have the property of attacking sericin and rendering it soluble so that it is easily removed from fibroin. An interesting, though not as well known, observation is that certain qualities of waste silk are capable of generating their own enzymes and degumming themselves, if immersed in water at room temperature for a period of several weeks. This method was said to produce very lustrous silk, but was not very popular because of the objectionable odors which were developed.

The following enzymes have been recommended for the degumming of silk, some of them in patents issued very recently: pepsin, trypsin, papain (either alone or activated with hydrocyanic acid or sodium thiosulfate), other proteases and bacterial proteolytic ferments, yeast enzymes, diastases from soy wort, and preparations obtained from the digestive organs of fish.

(Continued on page 303)

PROCEEDINGS OF THE
American Association of Textile Chemists and Colorists

MAY 21, 1934

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Members should notify the **Secretary** immediately of all address changes

The Preparation and Uses of

Starch Compounds*

By **JOHN T. GIBBONS**
Joseph Bancroft & Sons Co.

The Preparation and Uses of Starch Compounds

IN THIS talk, I want to tell you something about the various starches used in manufacturing processes, and how they can be modified and with what agents.

The starches in use can be divided into two classes:—

a—The starches obtained from cereals, such as wheat, rice, corn.

b—The starches obtained from roots, such as tapioca and potato.

The most important of these is corn starch because it is the cheapest in this country, which means it occurs in great abundance, and is easily manufactured. From it a great many useful products can be made, such as, modified or thin boiling starch, special gums, white and yellow dextrines, glucose, and corn sugar. Corn starch is used extensively in textile sizing, as a carrier and binder for the fillers, and also as a stiffening agent.

It is the cheapest and most effective agent for this work.

Tapioca, all of which is imported, was cheaper than corn for a time this year owing to foreign exchange but is now slightly above corn. It is a softer starch than corn and gives a more transparent jelly.

It has a very wide use in the textile industry for sizing and stiffenig textiles, and gives a more flexible, leathery handle than corn. Tapioca dextrine is the strongest of all dextrines, and is used almost exclusively as an adhesive on postage stamps, and as a gum for making envelopes. Tapioca starch is used for making a vegetable glue for

* Presented at meeting of Philadelphia Section, March 2, 1934.

veneering wood, and is the most satisfactory of the starches for this purpose.

Potato starch gives a clear translucent solution when boiled with water and is used where an opaque finish is undesirable, but it is always higher in price than the other starches and for this reason is used only on special finishes, that will stand the extra expense.

It is softer and smoother than corn or tapioca and is much brighter. It is often used in connection with borax to increase the brilliance of the finish. In this country potato starch is made from the smaller potatoes and those culled out as unfit for food. There is considerable variation in different lots owing to this fact. Potato starch made in Japan has been sold here, but it is not equal in strength to the American grade. The Russians are now offering a potato starch which is said to be superior to any other.

Sago starch is not on the market. Sago flour is used to some extent on black finishes, but its brown color, and the amount of dirt which is always present, prevents it from being used to any large extent. Sago flour has been bleached and washed free from dirt and in this form, it forms a clear colorless solution which is used to some extent to replace Gum Arabic. It also makes an excellent dextrine. Several gums made through bleaching and converting Sago are sold in this country, and are very satisfactory sizing agents, as the film is clear and will not give a grey tinge to a black shade.

Starches are an important factor in many manufacturing processes. They are used as food products, in the laundry business and the manufacture of shirts and collars as a stiffening agent. They are used in the rubber industry to remove the tack from shoe linings and to give a luster to raincoat fabrics. In textiles they are used as stiffening agents, and as adhesives, and printing gums, and for sizing warps to give the additional strength required in wearing.

In the manufacture of paper they are mixed with the paper pulp in the beater to disperse the size and cement the fibers. Converted starches have replaced glue to a large extent as a sizing for bond paper, to give a smooth finish, ink resistance, and a crackle to the paper.

The adhesives, gums, etc., made from starch are used for printing gums in the textile industry, as adhesives they are used in bookbinding, the manufacture of shoes, leather goods such as hand bags, and traveling bags. In the manufacture of trunks to paste on the linings, for bill posting, labeling, in the manufacture of oil cloth, carpets, paper bags and boxes, and veneering wood.

Glucose which is made from corn starch is used in the manufacture of confectionery, and as a sizing agent for textiles.

While this list I have given you is not complete by any means, it gives you an idea of the importance of starch as an article of commerce and how many industries make use of it.

All starches have the same formula, but each one has different physical properties. They differ in feel, appearance, water absorption power, and in the size and shape of the particles, and can be identified through these differences. The easiest way to identify a starch in a mixture is to wet a small amount with a little water, and rub the mixture out on a glass slide and examine it under a microscope. Microphotographs of the various starches have been published in books and magazines and by comparison with these pictures the starch under examination can be identified. If two or more starches are in the mixture, each one can be identified and the proportion of each one can be obtained roughly by counting the particles of each present and calculating the percentage.

To test the water absorption power of a sample, a small quantity is dried and boiled with a known quantity of water, and the paste obtained is compared for appearance and strength with known starches.

If one takes a sample of four starches, say, corn, tapioca, potato, and wheat, and rubs a little between the thumb and forefinger, the difference in the feel of each starch is very apparent, and after a little practice it is easy to identify a starch by feeling of it.

When starch is boiled with water a paste is obtained which has practically no flow and very little penetration on a fiber or fabric. A concentrated solution cannot be made owing to its high water absorption. It stays on the surface of the fiber and forms a caky surface.

If we want to size a warp to obtain additional strength which is needed in weaving, we want to penetrate to the center of the fiber with our size. We must use a converted or modified starch, in which the viscosity has been destroyed or reduced. When the viscosity of starch has been reduced or the fluidity increased, we call it a modified starch and the degree of modification is expressed in numbers such as:

90° thin boiling starch, or
60° thin boiling starch

which means that a certain number of c.c. of a starch solution of one concentration will flow from a pipette in a certain number of seconds.

Modification of Starches

Starches can be modified or converted in a great many ways and through a large number of agents.

Modification may leave the starches with most of its original physical properties, with the viscosity reduced, or if it is carried far enough, starch may be converted into sugar, so that a whole list of materials may be made, each one differing slightly from its predecessor, by continuing the action of an agent on the starch, all the way from starch to glucose.

A great many are now sold commercially and if a special starch is required with properties differing from those available, a product can be made to fit the use exactly.

Starches can be modified or converted by action of:—

(1)—Heat.

(2)—Heat and acids, such as:— (HCL (H_2SO_4 (Acetic

(3)—Alkalies, such as:— Caustic Soda.

(4)—Enzymes, such as:— Diastafor DeGomma Rapidase.

(5)—Oxidizing agents, such as:— Sodium Hypochlorite Sodium Peroxide.

(6)—Certain salts, such as:—$CaCl_2$ (Calcium Chloride) Zinc Chloride Ammonium Sulfate.

(7)—Heat and pressure, with a sudden release of the pressure after treatment.

Conversion by Heat and Acids

If starch is placed in a vessel and sufficient heat is applied, the starch is converted into a dextrine, but conversion by heat alone requires too much heat and too long a time, and gives an undesirable brown color to the dextrine. In practice a combination of heat and acid is used. A charge of starch is placed in a closed cylinder, which is rotated slowly, and a small percentage of acid is sprayed in, and thoroughly mixed into the starch. Heat is applied directly by means of steam in a jacket, or by gas or oil heat. A stirrer inside the cylinder scrapes the starch from the outer surface, so that lumps and cakes will not form through overheating. After the conversion takes place, which is tested by removing a sample and examining it to see how much starch remains, the charge is run out, cooled, sieved, ground, and allowed to stand in moist air to regain some of the moisture it has lost in the process.

The disadvantage of this method lies in the fact that the dextrine so formed has a brownish color.

To convert a starch by the wet process, the starch is mixed with water and acid and heated in a jacketed kettle until it is sufficiently converted. The amount of acid, the temperature used, and the time are all factors in this method, and by varying any or all of them different products can be formed. This method can be used to make pastes for general use.

After the conversion is carried far enough, the process can be stopped by adding enough alkali to neutralize the acid, and allowing it to cool. When borax is used to neutralize the acid a very adhesive paste is formed which has a good white color. The acid generally used in this process is sulfuric.

Acetic acid also acts on starch and partially acetylates it, to form an excellent sizing material, but the acetic acid process is more expensive, and it is very seldom used at the present time. The length of time required during acetic acid alone may run from 48 to 72 hours. In order to shorten the time a catalyst such as hydrochloric acid is used.

Starch is placed in an acid-proof vessel, and a mixture containing 30% acetic acid, and 1.5% hydrochloric acid is run in. The mass is agitated continuously and the temperature is kept below 38° C. for about 48 hours. Then the acid is removed by decantation and filtration, and the product washed to remove all the acid, and then dried. The product obtained by this process contains about 7 to 8% of acetylated starch, and is quite different from any thin boiling starch.

Conversion by Oxidizing Agents

A modified starch can be made mixing the starch with a bleaching solution, either sodium hypochlorite, or Ca. hypochlorite, agitating at a temperature below the swelling point of starch until the conversion takes place, then filtering, washing to remove any traces of the hypochlorite, and then drying.

For this process a solution of sodium hypochlorite of 10° Tw. is made up and mixed with an equal weight of starch. Heat is given off from the reaction taking place and the temperature must be kept below the swelling point of the starch.

The mixture can be left over-night before washing to remove the hypochlorite. Two actions take place, one a bleaching of the starch, and the other a modification. This process can be used to convert sago flour and wash it, the product of which is a white clean sizing material.

Conversion by Enzymes

The action that enzymes have on starch is well known, because they are commonly used to remove starch from fabrics before boiling and bleaching. It is also possible to modify a starch to any desired consistency by heating a mixture of starch, water and an enzyme at 150° to 160° F. until the right point is reached, and then raising the temperature rapidly to the boiling point, which destroys the enzyme and stops the action. This process is now being used to make warp sizing in the mills and the size so made is cheaper, does not clog the size pipe because its fluidity is permanent, and gives better penetration than the thin boiling corn generally used. The warps sized with it are more easily conditioned, because this size absorbs water faster than a thin boiling corn.

It is possible to get a very highly concentrated solution by this method, which is often desirable. Ordinarily if we want to get a mixture containing 8 pounds of dry material to a gallon of water the only size we can use is a corn dextrine, and that will give a mixture that has considerable body when cold. By converting a starch with an enzyme we can use 10 or 12 pounds of starch to the

gallon of water and regulate the time of the conversion to obtain practically any fluidity.

Conversion by Alkali

An alkali, such as caustic soda will act on starch to convert it into a stringy adhesive paste. To accomplish this, starch and water are placed in an agitator and about 10% of caustic soda on the starch is added. The mass is agitated continuously, as it has been shown that the physical agitation reduces the stringy quality of the paste and the longer the agitation is continued, the greater the effect.

This principle has been used in preparing a dry paste, which consists of a mixture of starch and caustic soda, both in the dry form. When water is added to the powder and the mass is agitated the action of the caustic starts and the starch is converted.

It has been further discovered that if the starch is agitated with acid for some time before the treatment with alkali that the viscosity is reduced and the paste is smoother. A physical change takes place and the size of the starch agglomerate is reduced.

Starch, water and acid are placed in a vessel and agitated, at room temperature. After this action has gone on from four to six hours, the acid and water are removed, and the starch is dried. It has been found that the same amount of acid is present at the end of the agitation, as was present at the start, but on boiling the starch with water we find the viscosity is reduced.

If the starch is now treated with caustic soda the resulting vegetable glue is smooth, spreads easily and is very adhesive.

In this condition it is strongly alkaline, and if this is objectionable, the alkali can be neutralized and the glue left on the acid side. This improves the color also.

Tapioca is used for this glue entirely, as it forms the best product in terms of strength, smoothness and cost. This glue is the one used in veneering wood, and the process is patented by the Perkins Glue Co. These patents have been contested in court and I believe the litigation is still going on.

Conversion by Salts

If starch is heated with calcium chloride, zinc chloride, ammonium thiocyanate and numerous other compounds, in the presence of water, the starch is converted and forms an adhesive paste. This method is used for special pastes when certain properties are desired, that cannot be obtained from the ordinary dextrines or gums.

Conversion by Heat and Pressure

Starch is heated in a closed vessel under pressure and after a time the pressure is suddenly released. The moisture which is contained in the starch has been changed to vapor by the heat, and when the pressure on this vapor is suddenly released it expands, and bursts the granules of starch. The product of this process is soluble in cold water and forms an adhesive paste. This is the method used to prepare food "shot from the guns."

Waterproofing of Starch

Starch would be a much more valuable material if we could make it waterproof. Its affinity for water is one of its characteristics, and it is difficult to overcome this property completely. However, starch can be waterproofed or rendered insoluble in water to a considerable extent, but the resulting product is hard to handle, especially in applying it to a fabric.

I have tried waterproofing the starch after it had been applied to the fabric, but I know of no way of doing this without injuring the fabric. If we heat a mixture of starch, water and lime, we obtain a material which is only slightly soluble in water. Barium hydroxide can also be used to obtain this effect. The material is spongy and cannot be spread out smoothly. This effect can be obtained indirectly also, by first treating the starch with calcium chloride, and then adding a solution of caustic soda, to set free the calcium hydroxide to act on the starch, and secure a smooth even result which has considerable use.

Discussion

Dr. Powers: I would like to ask about the honey locust bean, is that a competitor of starch?

Mr. Gibbons: I never did consider it a competitor of starch.

Dr. Powers: As a competitor to starch, has any work been done in modifying that?

Mr. Gibbons: It really isn't a competitor of starch; it is out of the starch field. It is considerably more than starch in price.

Dr. Powers: I was wondering if any work had been done in modifying the honey locust bean so that it would have more application and wider use as a substitute for starch.

Mr. Gibbons: No, I don't know of anything that has been done on that.

Mr. Bruce: Mr. Gibbons, could any of those modification processes that you mention for modifying starch be used directly in the finishing mix and done that way?

Mr. Gibbons: Yes. That can be done if you follow that procedure. That is what is being done on warp sizing, and, of course, finishing would follow that way. A number of southern mills use it for warp sizing and they are treating corn starch with an enzyme. They are saving money doing it and getting a better product.

Mr. Bruce: Can the every-day textile mill control that conversion accurately enough to get a result that is comparable to one after another?

Mr. Gibbons: I think so. Most of the mixing room men are pretty expert in doing that sort of thing. They might not get the results the first time, but they can do it.

Mr. Bruce: I note that you mention time is a very important factor in those things so that running it a little too long would give you something entirely different.

Mr. Gibbons: Not entirely different. You have considerable leeway; it is not a matter of seconds. I guess I did mislead you on that. A few seconds would not make a lot of difference.

Mr. Bruce: That acetic acid mixture that you speak of—that isn't possible to do, is it?

Mr. Gibbons: It certainly is.

Mr. Bruce: Right in the finishing mix or is it done in separate processes?

Mr. Gibbons: Separate processes. That would require a fair amount of equipment you wouldn't find in an ordinary mill.

NINTH MEETING RAYON SUB-COMMITTEE

THE ninth meeting of the Rayon Sub-Committee was held in the Chemists' Club in New York City at 8:00 P.M. immediately following the usual dinner at 6:30 P.M.

The meeting was well attended — eighteen members and guests being present.

Two pending matters, revision of the Committee's publication "Rayon Identification" and the final corrections of the tentative publications "Short Cut Tests for Determining Certain Properties and Causes of Faults Observed in Rayon Yarns and Fabrics" were disposed of at the meeting both being released for publication at an early date in the proceedings of the Association.

A Sub-Committee of the Sub-Committee was chosen to prepare for submission to the Sub-Committee proper a survey of the current Ladies Dress Rayon Crepe problem together with a definite procedure for attack on certain phases of this problem and an estimate of the cost of the proposed research, in order that the necessary funds might be raised from the Rayon Industry and other interested sources.

It was decided that the problem of evolving a method of determining the shrinkage of rayon fabrics including crepe fabrics and circular knitted fabrics should be undertaken by the Committee. The newest member of the Committee, Mr. Zametkin, volunteered to devote a portion of his time to the solution of this problem.

Considerable time was spent upon a detailed discussion of the Rayon Symposium that is to be presented by the Committee at the December Annual Meeting of the Association.

It was decided that the presentation will be limited to three papers, each somewhat detailed and each dealing with current up-to-the-minute problems.

The meeting was adjourned at 11:30 P.M.

CORRECTION

In my remarks made at the Annual Meeting last December in connection with the work of the Light Fastness Committee of the A.A.T.C.C. I stated that the British Wool Industries Research Association had developed a set of standards of light fastness which had been officially adopted by that organization.

J. B. Atkinson, the Secretary of the Society of Dyers and Colourists has brought to my attention the fact that the work done in England in connection with standards of light fastness was initiated by the Society of Dyers and Colourists and that all of the work has been carried out entirely under their auspices. Arrangements were made by that Society for the necessary cloth to be dyed, exposed and tested and it was in this connection where the Wool Industries Research Association played a most important part.

The Report of this work on Standardizing the Methods of Testing the Fastness of Dyed and Colored Materials, which is the culmination of many years labor is now being finally edited and will be published by the Society of Dyers and Colourists in the course of a month or two.

We are glad to note from Secretary Atkinson's letter that the work of standardizing the methods of testing the fastness of dyed and colored materials is progressing so well and it is hoped that if these methods are published that our own Research Committee will study these methods carefully with the object in mind of eventually being able to formulate a set of standards which will be internationally uniform.

L. A. Olney,
Chairman Research Committee.

MEETING, LOWELL TEXTILE INSTITUTE STUDENT SECTION

On May ninth, Dr. Olney addressed the Lowell Textile Institute Section on the value of the association to its members in the textile industry. Kenneth E. Leslie was elected Chairman, and Chester M. Kopatch, Secretary.

Respectfully submitted,

Chester M. Kopatch,
Secretary

Proceedings of the American Association of Textile Chemists and Colorists

Determination of Inorganic Salts in Sulfonated Oils

Report No. 3 of the Subcommittee on Sulfonated Oils*

By RALPH HART, Chairman

Inorganic Impurities

INCIDENTAL to manufacture or through admixture, commercial sulfonated oils may contain various amounts of inorganic salts, such as alkali chlorides, sulfates, and less frequently acetates, carbonates, etc. The determination of inorganic sulfate in sulfonated oils is part of the regular procedure for finding organically combined sulfuric anhydride by the barium sulfate methods, but the other inorganic salts are practically always included with the "undetermined," largely because no satisfactory direct method of analysis for inorganic salts has been described. A satisfactory method would not only round out the analysis of a sulfonated oil but would also be of great assistance in grading or classifying such products. Thus, in connection with Commercial Standards CS43-32 of the Bureau of Standards,[3] the Committee was in favor of grading sulfonated oils by subtracting from 100% the sum of the moisture and inorganic salts, but this method had to be abandoned because the methods for determining inorganic salts were unsatisfactory. Moreover, with the amounts of inorganic salts, moisture and organically combined sulfuric anhydride determined, it is a comparatively simple matter to decide which of the various methods proposed for the determination of fatty matter in sulfonated oil is likely to be the most reliable.

In the present report of the Subcommittee on Methods of Analysis and Grading of Sulfonated Oils, a standard method for the determination of inorganic sulfates and chlorides in sulfonated oils is proposed as well as an optional method for alkali carbonates and acetates. In the previous two reports,[5, 9] standard methods for moisture and organically combined sulfuric anhydride, respectively, have been recommended. The membership of the committee remains the same as when first appointed and consists of the following:

Ralph Hart, 1440 Broadway, New York City, Chairman.
J. Andrew Clark, Dutchess Bleachery, Inc., Wappingers Falls, N. Y.
Geo. P. Feindel, Union Bleachery, Greenville, S. C.
Stephen K. Ford, Marden-Wild Co., Somerville, Mass.
Arthur L. Fowler, Jr., American Cyanamid & Chemical Corp., Woodbridge, N. J.
A. H. Grimshaw, N. C. State College, Raleigh, N. C.
C. P. Gulick, National Oil Products Co., Harrison, N. J.
R. A. Pingree, U. S. Finishing Co., Providence, R. I.
I. Silverman, L. Sonneborn Sons, Inc., Belleville, N. J.
W. S. Williams, Mt. Hope Finishing Co., North Dighton, Mass.

Others who have submitted analytical data are the following:

M. B. Hart, The Hart Products Corp.
E. Segessemann, National Oil Products Co.
Fred R. Tripp, Mt. Hope Finishing Co.
A. Winograd, L. Sonneborn Sons, Inc.

It may be of interest to note that since the last report of this committee, the American Oil Chemists' Society has appointed a committee with a similar scope, namely, the "A.O.C.S. Committee on Methods of Analysis and Grading of Sulfonated Oils with Particular Reference to the A.A.T.C.C. Modifications." A number of our members are serving on this committee, including J. A. Clark, R. Hart, A. H. Grimshaw, H. H. Mosher, R. A. Pingree, W. H. Tiffany and R. Wechsler.

Available Methods

Herbig[10] determines inorganic sulfate by dissolving the sample in ether, washing with a saturated solution of sodium chloride, and precipitating the sulfate in the wash water with barium chloride. To overcome troublesome emulsions, which highly sulfonated oils tend to form, he replaces one-third of the ether with benzene. If emulsions still persist, Herbig dissolves the oil in absolute alcohol, filters, dissolves the residue in hot water and determines the inorganic sulfate in the filtrate. Nishizawa[12] attributes these emulsions to the comparatively easy solubility of the normal or sodium salt of sulfonated oils in concentrated sodium chloride solution, and further states that the compound is salted out with considerable difficulty even in the presence of solid sodium chloride. On the other hand, the monosodium salt is practically insoluble in saturated sodium chloride solution. Accordingly he modifies the Herbig method by first converting the compound to the monoso-

*A portion of this report has already been published in the May 15, 1934, Analytical Edition of *Ind. and Eng. Chem.*

dium salt with hydrochloric acid, using methyl orange as the indicator.

Burton and Robertshaw[2] determine total inorganic salts by digesting the dehydrated sample with alcohol or alcohol-ether, filtering, dissolving the residue in hot water, and determining the solids in the filtrate by evaporation. The inorganic salts, they state, are not completely precipitated with alcohol with many sulfated oils. Some tests made in this laboratory indicate that the Burton and Robertshaw method may yield good results with sodium sulfate but is not satisfactory with sodium chloride using 95% alcohol as one of the solvents. The results on a sample, in which the inorganic salts contained about 90% sodium chloride and the rest sodium sulfate, were as follows:

Solvent	*Inorganic Salts* Added %	Found %
2 parts ethyl ether and 1 part 95% alcohol	7.84	6.32
2 parts ethyl ether and 1 part absolute alcohol	7.84	7.52

Difficulties may also be experienced with the alcohol-ether solvent in the case of dehydrated oils containing at the same time large amounts of fatty glycerides or mineral oils, and neutral soap. The former are sparsely soluble in alcohol and the latter in ether.

Wizöff or Herbig Method

In the official method of the Wissenschaftliche Zentralstelle für Öl-und Fettforschung (Wizöff),[14] a mixture of 40% ether and 60% amyl alcohol is used as solvent for the oil, otherwise the procedure is the same as the Herbig benzene-ether method. The procedure for this method is as follows:

Determination of Inorganically Combined Sulfuric Acid. 5 to 7 g. of oil is shaken up in a separatory funnel with 10 c.c. saturated sulfate-free common salt solution, 10 c.c. ether, and 15 c.c. amyl alcohol; the clear salt solution is separated from the ether and then washed three times with 10-20 c.c. of conc. salt solution. The combined salt solutions are made up to 25 c.c., 1 c.c. of concentrated HCl (D-15 = 1.19) is added, and the sulfuric acid estimated by precipitating at the boil with barium chloride solution in the usual way.

The A. L. C. A. Method

Another method for total inorganic salts is that of Hart.[5, 6] According to this method, total nonvolatile inorganic salts are determined indirectly from the ash, fixed alkalinity, and organically combined sulfuric anhydride in the sample. This procedure is also the official method of the American Leather Chemists Association (A.L.C.A.)[1] It yields reliable and accurate results, except in the presence of ammonium salts and sodium acetate; the former is volatilized during the ignition and the latter changed to the carbonate. Moreover, the formulas are calculated for sodium salts and in the presence of potassium it becomes necessary to determine the metallic elements. The following are the details of this method:

Ash—Weigh accurately from 5 to 10 grams of the sample into a crucible or dish, ignite gently, allowing the oil to burn, and then complete the incineration at a dull red heat until all the carbon is burned off. Cool and weigh.

Calculations—The inorganic, nonvolatile, stable salts are given by these formulas:

Case I—When $(A_f - 7S)$ is negative,

$$\% \text{ Inorganic Salts} = \% \text{ Ash} - 0.1268\ (A_f + 7S)$$

Case II—When $(A_f - 7S)$ is positive,

$$\% \text{ Inorganic Salts} = \% \text{ Ash} - 0.0946\ (A_f + 11.77S)$$

Where A_f represents the fixed alkalinity, expressed in mgms. KOH per gram of sample, and S the per cent organically combined SO_3.

These formulas are based on the following reactions:

(a) The sulfonated radical, in the absence of soap or other alkali, is converted into equal parts of sodium sulfate and sulfuric anhydride upon ashing, thus:

$$2\,ROSO_3NA \longrightarrow Na_2SO_4 + SO_3 + \text{etc.}$$

(b) In the presence of soap or other alkali part or all of the liberated SO_3 may be bound by the former, depending upon the amount of alkali in the sample. The reaction is as follows:

$$2\,RCOONa + SO_3 \longrightarrow Na_2SO_4 + CO_2 + \text{etc.}$$

(c) Soap or other alkali in excess of the amount necessary to combine with all the liberated SO_3 is changed upon ashing into sodium carbonate. The derivation of these formulas are given elsewhere.[4, 5]

Solvent Method for Alkali Sulfates and Chlorides

It will be noted that in the "Wizöff" method there is no provision made for estimating chlorides, or sodium, potassium and ammonium ions of the inorganic salts. The shortcomings of the A.L.C.A. method have already been mentioned. After considering these and other objections, the committee was of the opinion that a method would be desirable which would determine the sulfates and chlorides directly and in the form in which they are present in the original oil. It therefore decided to concentrate its work on solvent methods. Besides the literature already mentioned, it is also to be noted that McBain and McClutchie[11] found that soda soaps dissolve and form jellies in nonpolar solvents, such as benzene, xylene, etc., and also that sodium oleate and stearate exhibited no signs of gelation in acetone or chloroform.

The writer finds that sulfonated oils, particularly when neutralized and dehydrated, dissolve in ethyl ether or carbon tetrachloride with great difficulty, giving practically nonfilterable solutions. However, the dehydrated oil becomes miscible with these solvents upon the addition of oleic acid. Taking advantage of this fact and also of the practically negligible solubility of sodium chloride and sulfate in ether or carbon tetrachloride, a method for determining these salts in sulfonated oils has been developed. Details of procedure are given at the close of this report. It consists essentially of dehydrating the sample in the presence of oleic acid, dissolving in ether or carbon tetrachloride, and filtering off the residue. The use of oleic acid[7] as a coupling agent and for liquefying gels of soap and solvent is well known. Oleic acid or its equivalent is invariably used in the determination of water in soap by the distillation method[4] to prevent foaming and to keep the solvents liquid. Carbon tetrachloride, suggested by R. A. Pingree, was found to be the best of several solvents tested.

Discussion of Solvent Method

The ordinary sulfonated oil was found to be stable when heated as outlined as long as there was some soap present, even as little as 2.0% on the weight of the sample. There was also no reaction between the salt and the free fatty acids upon heating. This was shown by the fact that the alkalinities of a sample containing salt were identical before and after heating. If a reaction did occur, soap would form and the alkalinity of the sample would be correspondingly increased. Evidently alkali added to stabilize the oil will not affect the result, provided it is all converted into soap. Oleic acid is added to the carbon tetrachloride in the first washings in order to prevent precipitation of soap on the residue.

The determination of the dried as well as the ignited residue is suggested in order to detect the presence of insoluble fatty matter or ammonium salts, or incomplete washing. Ignition of the well-washed residue, even in the absence of ammonium salts, invariably charred slightly and showed a loss of 0.1 to 0.2%. This loss probably represents fatty matter adsorbed by the salts which is difficult to remove even upon prolonged washing. Where the loss upon ignition is greater than 0.3%, it is probably due, in the absence of ammonium salts, to incomplete washing of the residue; in which case it is necessary to repeat the analysis with more thorough washing. Evidently the error due to adsorbed fatty matter is reduced to a minimum by ignition. For this reason and the fact that the "ignited" rather than the "dried" results check more closely with the ash and barium sulfate methods, it is recommended that in the absence of ammonium salts, the "ignited" residue be reported as the final results. The residue, of course, may be analyzed for its constituents by the usual methods for inorganic analysis. It is to be noted that this method yields the alkali salts in the form actually present in the original oil.

Experimental

In Table I are listed sulfonated products, containing sodium or ammonium sulfate or sodium chloride, that were analyzed for their salt content by the solvent method and, in a few cases, also by the Herbig or barium sulfate method. The figures represent the averages of several analyses. The differences in the amount of sodium sulfate by the two methods for samples 1 and 2, given in the last column, were 0.04% and 0.08%, respectively, and for sample 3 containing ammonium sulfate the difference was 0.21%. Sample 4 showed 4.93% of sodium chloride by the solvent method, compared with 4.91% added. The loss upon ignition of sample 8 was considerably larger than with any of the other samples. Sample 8 is a special product, brought on the market within the past few years as a detergent, and is supposedly a condensation product of a fatty acid and a sulfonated amino compound. Whether the greater loss in this case is due to fatty matter insolu-

TABLE I
Inorganic Sulfates and Chlorides in Sulfonated Oils

Sample	Description	Nature of Salt	Inorganic Salt: Solvent Method Dried %	Inorganic Salt: Solvent Method Ignited %	Inorganic Salt: Difference %	Inorganic Salt: Barium Sulfate Method %	Inorganic Salt: Difference %
1	Sulfonated castor oil	Na_2SO_4	0.92	0.83	0.09	0.79	0.04
2	Sulfonated castor oil	Na_2SO_4	6.72	6.64	0.08	6.56	0.08
3	Sulfonated castor oil	$(NH_4)_2SO_4$	8.52	...	...	8.73	0.21
4	Sulfonated castor oil	NaCL + Na_2SO_4	5.00	4.93[b]	0.07	...	...
5	Highly sulfonated oil[a]	Na_2SO_4	2.92	2.84	0.08	...	...
6	Sulfonated tallow	Na_2SO_4	6.41	6.29	0.12	...	...
7	Sulfonated mineral oil	Na_2SO_4	0.25	0.22	0.03	...	...
8	Sulfonated detergent[c]	NaCl + Na_2SO_4	18.52	17.75	0.77	...	...

[a] 93% sulfonated.
[b] Added 4.91%.
[c] Commercial sample of a condensation product of a fatty acid and a sulfonated amino compound.

ble in the solvent or to insufficient washing was not further determined. Ignition of the dried residues was accompanied by slight charring, apparently due to adsorbed fatty matter. In the remaining samples listed in Table I, the reliability of the solvent method is indicated by the small differences in weight between the "dried" and "ignited" residues. That soaps of saturated fatty acids do not interfere with the method is shown by sample 6, representing a neutralized sulfonated tallow, which upon ignition lost only 0.12%.

TABLE II
Committee Results of Inorganic Salts in Sample C

	Inorganic Salts (Na_2SO_4)							
	Solvent Method						Ash Method	Barium Sulfate Method
	Dried			Ignited				
	I %	II %	Av. %	I %	II %	Av. %	Av. %	Av. %
Clark	0.89	0.99	0.92[a]	...	...	...	0.73	0.80
Ford	1.30	1.52	1.39[a, b]	1.18	1.35	1.26[a, b]	0.79	...
Fowler	0.80	0.78	0.79	0.76	0.72	0.74	0.76	0.75
Gulick	1.09	1.09	1.09	0.81	0.81	0.81	0.76	0.76
Hart	0.95	0.89	0.92	0.85	0.80	0.83	0.83	0.79
Pingree	0.83	0.84	0.84	0.78	0.78	0.78	0.81	0.76
Silverman	0.82	0.97	0.93[a]	0.71	0.77	0.75[a]	0.82	0.85
Williams	1.07	1.06	1.07	0.93	0.96	0.95	0.71	0.71
Grand average			0.93			0.81	0.78	0.78
Greatest difference			0.30			0.21	0.12	0.14
Average deviation from mean			0.08			0.05	0.04	0.08

[a]More than two determinations.
[b]Omitted from calculations.

TABLE III
Committee Results of Inorganic Salts in Sample C-1

	Inorganic Salts (Na_2SO_4)							
	Solvent Method						Ash Method	Barium Sulfate Method
	Dried			Ignited				
	I %	II %	Av. %	I %	II %	Av. %	Av. %	Av. %
Clark	6.91	7.09	7.00	...	...	...	6.53	6.41[a]
Ford	7.06	7.18	7.11[a]	6.86	7.02	6.95[a]	6.81	...
Fowler	6.85	6.90	6.87	6.73	6.75	6.74	6.16[b]	6.22
Gulick	6.91	6.91	6.91	6.64	6.68	6.66	6.03[b]	6.03[b]
Hart	6.74	6.70	6.72	6.65	6.62	6.64	6.66	6.56
Pingree	6.80	6.82	6.81	6.68	6.67	6.68	6.64	6.55
Silverman	6.79	7.10	6.96[a]	6.46	6.69	6.55[a]	6.65	6.90
Williams	6.89	7.05	6.95[a]	6.80	6.98	6.86[a]	6.91	7.16[b]
Grand average			6.92			6.73	6.70	6.53
Greatest difference			0.39			0.40	0.38	0.68
Average deviation from mean			0.09			0.11	0.11	0.17

[a]More than two determinations.
[b]Omitted from calculations.

TABLE IV
Summary of Committee Results of Inorganic Salts in Samples C and C-1

	Inorganic Salts (Na_2SO_4)					
	Sample C			Sample C-1		
	Solvent-Ignition Method %	Ash Method %	Barium Sulfate Method %	Solvent-Ignition Method %	Ash Method %	Barium Sulfate Method %
Grand averages, per cent	0.81	0.78	0.78	6.73	6.70	6.53
Greatest difference, per cent	0.21	0.12	0.14	0.40	0.38	0.68
Average deviation from mean, per cent	0.05	0.04	0.08	0.11	0.11	0.17
Difference between solvent and ash method, per cent	0.03			0.03		
Difference between solvent and barium sulfate method, per cent	0.03			0.20		

Committee Results

Committee Sample C and C-1 were submitted to the committee for the determination of inorganic sulfate by the following three methods: (a) ash, (b) barium sulfate, and (c) the new solvent method. Sample C was a commercial sulfonated castor oil, and sample C-1 was the same oil with the addition of about 7.0% anhydrous Glauber's salt. The results are given in Tables II and III. In Table IV a summary of the results are given from which it will be noted that the difference between the solvent and ash method for samples C and C-1 was 0.03% in each case, and between the solvent and the barium sulfate method, 0.03% and 0.20%, respectively. It is thus evident that the solvent-ignition method compares favorably with the two official methods and possesses the additional advantages that the manipulations are less difficult, ammonium salts as well as chlorides are estimated, and the sulfates and chlorides are determined not only directly but also in the form in which they are present in the original sample.

Determination of Sodium Acetate and Sodium Carbonate

The solvent method unfortunately cannot be used in the presence of sodium acetate or carbonate, since these salts react with the free fatty acids to form soap when the oil is dehydrated. It has also been found that solid sodium acetate, thrown out of a neutralized oil during dehydration, reacts quantitatively with the excess oleic acid added to the solvent to effect solution of the oil. This reaction takes place even at temperatures below the boiling point of the solvent. Trotman[13] made experiments to determine whether different proportions of oleic acid are able to decompose sodium acetate completely when distilled with steam. When only small quantities were used, decomposition was practically complete, the acetic acid in the distillate corresponding to the whole of oleic acid taken. When larger quantities of oleic acid were taken, only a portion was recovered as acetic acid.

A method available for acetate is to determine the acetic acid by steam distillation with phosphoric acid. However, the volatility of fatty acids[13] with steam and the presence of carbonate may interfere with the accuracy of this method. Alkali carbonates may be determined as in soap analysis, by taking advantage of their insolubility in alcohol. The dehydrated sample is digested in absolute alcohol or with a mixture of absolute alcohol and ether, and the residue after filtration is washed free of titratable salts, such as sodium acetate, etc. The residue is then dissolved in hot water and the carbonate determined in the filtrate by titration.

More rapid and convenient titration methods for both alkali carbonates and acetates have been developed in this laboratory and seem to be accurate within 0.2%. Since sodium acetate cannot be accurately titrated with the usual indicators because of the free acetic acid, it becomes necessary to remove the acid either by volatilization or by washing. The titration methods are based on the following findings. Total fatty matter in a sulfonated oil may be extracted quantitatively without decomposition with ether over an acidified solution of salt; acetic acid dissolved in ether may be extracted practically completely by proper washing with salt solution; sodium acetate may be quantitatively decomposed, liberating acetic acid, by heating with excess oleic acid at 123° to 125° C; and a sulfonated oil containing soap may be heated for 30 minutes at 123° to 125° C. with practically no decomposition of the organically combined sulfate group. The titration method, which is given at the end of this report, is varied somewhat, depending upon whether or not the sample contains carbonates, acetates, or both.

Experimental

Three samples of oil, containing known amounts of sodium carbonate, sodium acetate, and a mixture of the two, were analyzed according to the titration methods and the results are given in Table V. Sample C-2 was made by mixing a titrated solution of sodium carbonate with an equal amount of a neutralized sulfonated oil of known alkalinity. Sample C-3 was a mixture of a titrated solu-

TABLE V
Alkali Carbonates and Acetates in Sulfonated Oils by New Titration Methods

	Sample C-2 Mg. KOH/g.	Sample C-3 Mg. KOH/g.	Sample C-4 Mg. KOH/g.
Alkalinity as free fatty acids (A_f)			
Found	0.00	4.5	0.00
Calculated	0.00	4.4	0.00
Alkalinity as soap (A_s)			
Found	34.0	28.4	34.2
Calculated	33.4	29.0	33.4
Total alkalinity (A_t)			
Found	59.2	49.9	56.9
Calculated	59.8	49.6	56.9
Alkalinity as soap (A_s) and as carbonate (A_c)			
Found	...	...	45.9
Calculated	...	...	46.6
Sodium carbonate	%	%	%
Found	2.38	...	1.11
Calculated	2.49	...	1.25
Difference	0.11	...	0.14
Sodium acetate			
Found	...	3.14	1.61
Calculated	...	3.01	1.51
Difference	...	0.13	0.10

tion of acetic acid exactly neutralized with caustic soda, and an equal amount of a sulfonated oil with known alkalinity and acidity. Sample C-4 was a mixture of 25% each of the carbonate and acetate solutions, and 50% of the neutralized sulfonated oil. The results in Table V are the averages of several determinations in each case. It will be noted that the amount of sodium carbonate found in sample C-2 differed from the calculated value by 0.11%, that the difference in sodium acetate for sample C-3 was 0.13%, and the difference in sodium carbonate and sodium acetate for sample C-4, were 0.14% and 0.10%, respectively. The accuracy of these results is well within the experimental error and is considered satisfactory for the analysis of sulfonated oils.

It seems that the samples containing the carbonate liberate fatty acids upon standing, which react with the alkali, giving lower results for the amount of carbonate present in the samples than when they were first made up. The amount of acetate, however, is not affected. The results of the analyses of the three samples after standing for about one month were as follows:

	Sample C-2	Sample C-3	Sample C-4	
	Sodium Carbonate	Sodium Acetate	Sodium Carbonate	Sodium Acetate
	%	%	%	%
Pingree	2.04	3.24	1.00	1.50
Hart	2.07	3.18	1.05	1.59
Calculated	2.49	3.01	1.25	1.51

The committee believes that alkaline salts, since they occur only in special products and then only infrequently, should be found by difference or, if a direct estimate is desirable, they may be determined by the titration methods, outlined at the end of this report.

Comments and Suggestions:

The following comments and suggestions were received from the members of the committee:

ARTHUR L. FOWLER: I think that the solvent method is a very quick way of determining all of the impurities in sulfonated oils. A Gooch crucible, suggested by Mr. Segessemann, was found to be a vast improvement over filter paper. It might be interesting to note that the salts retained in the crucible will have a brownish color when covered with carbon tetrachloride, unless free from oleic acid or soap. When the salts are white the sample has been washed free of oleic acid or soap. Even then they are not entirely free of organic matter; hence, it is best to ignite the crucible in order to be sure that no organic matter is weighed. Carbon tetrachloride varies a great deal, some of it is not fully chlorinated. Also on heating, if any water is present it will hydrolyze and develop HCl which would be detrimental. Therefore I think rigid specifications should be set up for this solvent before being used as a reagent.

C. P. GULICK: (1) *Solvent Method*—This method appears to be the most convenient, the simplest and also the most generally applicable, since it determines the chlorides as well as the sulfates and irrespective whether they are present as sodium or potassium salts. In the first step of the procedure, i.e., the dehydration of the oil, I would suggest the use of a beaker "extra tall" form rather than a crucible. This would eliminate the trouble and a possible source of error of transferring the oil from crucible to beaker. A Gooch crucible in place of the filter paper would in my opinion simplify the technique.

(2) *Ash Method*—The application of this method would have to be limited only to oils free from ammonium salts. Furthermore, the calculations are correct only for oils that have been soda-neutralized and finished. For potash neutralized oils the necessary correction would have to be made and for oils containing both potash and soda, this procedure would become an impossible task for the ordinary laboratory.

(3) *Barium Sulfate Method*—Also this method would have a limited application. It could be used only for oils where the inorganic salts are known to consist entirely of sodium sulfate. It would necessitate correction for oils containing potassium and ammonium salts and would give fictitious results if impurities were present in the form of chlorides.

RALPH HART: The time lost in preparing a Gooch crucible may be saved by using a counterpoised filter paper, but the slower filtration through the latter may counterbalance the gain. The use of ether in this laboratory as well as by several members of the committee gave equally as good results as with carbon tetrachloride as the solvent. The latter, however, gives a clearer and a more satisfactory solution to filter.

The error due to the ash from the inorganic matter chemically combined with the adsorbed fatty matter is apparently very small and is probably never greater than one-half of the loss upon ignition. Since the per cent inorganic salts in the average sulfonated oil is rather small, comparatively small amounts of fatty matter left in the residue may cause a larger error in the result, especially in the presence of ammonium salts when the residue is not ignited. It is, therefore, deemed advisable to specify very thorough washing of the residue.

R. A. PINGREE: Ether evidently is a very poor solvent for both soap and the sodium salt of the sulfonated oil. The insolubility of the latter seems to be overcome, in part at least, by the use of oleic acid, but this does not overcome the insolubility of the soaps. In searching for a more satisfactory solvent to use in this method, I have found that carbon tetrachloride seems to fill about all the requirements. It is a solvent for soaps, sulfonated oils, raw oils, etc., but will not dissolve any of the inorganic salts which are likely to be found in a sulfonated oil. In

some work I did several months ago on the solubility of potassium oleate in organic solvents, I found that it was soluble in carbon tetrachloride to the extent of more than 10%. Carbon tetrachloride also has a boiling point sufficiently high to permit warming and consequently more rapid solution of the soaps, etc.

The following experiments were made to find the minimum amount of washing necessary to free the residue from impurities. I ran two determinations with Committee Sample C-1. I followed the regular procedure until the dehydration of the sample was complete. 50 cc. of carbon tetrachloride was then added, heated to approximately 60° C. and filtered thru a Gooch crucible. After allowing it to drain, the remaining residue was rinsed from the beaker into the crucible, using 25 cc. of hot CCl_4 (+ 2% oleic acid) from a small wash bottle. Allowing it to drain again, I then washed the residue in the crucible with 10 cc. hot CCl_4 (+ 2% oleic acid), followed by two 10 cc. portions of ether, allowing each washing to drain before adding the next. I considered this to represent a minimum amount of washing. The crucible was dried in the oven at 120° C. for 1 hour, cooled in a desiccator over $CaCl_2$ and weighed. The crucible was then replaced in the suction funnel and washed further with three 15 cc. portions of boiling CCl_4 (+ 2% oleic acid), five 15 cc. portions of boiling CCl_4 (without oleic acid), and two 15 cc. portions of ether. The crucible was then dried at 120° C. for 1 hour, cooled as before and weighed. The date obtained is given below.

Per Cent Salt

Sample	*Minimum Washing*	*Maximum Washing*	*Ignited*
I	6.71	6.71	6.63
II	6.77	6.75	6.66

Judging from these figures, it seems quite evident that the extra precautions in washing are quite unnecessary. There is evidently a small amount of organic matter present in the oils which is insoluble in CCl_4 and ether. I suggest that after transferring the residue from the beaker to the Gooch crucible, the washing procedure consist of two 15 cc. portions of boiling CCl_4 containing 2% oleic acid, two 15 cc. portions of boiling CCl_4 without oleic acid, and lastly, two 15 cc. portions of ether. I think that this amount of washing will be more than sufficient to remove the last trace of soluble matter from residue.

The close agreement of all results obtained by the Solvent method was quite remarkable. I find, however, even after prolonged washing, there still remains a small amount of organic matter with the precipitated salt. The error introduced by this organic matter is quite small and I believe may be considered negligible. This error seems to be a constant with each individual oil and amounted to + 0.055% in the case of sample C and + 0.14% in sample C-1. I am fully in favor of recommending this method to the Association.

Titration Methods: I have found these methods for sodium carbonate and sodium acetate to work very nicely on the samples submitted. However, they would only be of value where qualitative tests have shown no interfering substances, such as ammonia, borax, sodium phosphate, etc. to be present. In the presence of any of these substances, the methods would obviously have to be modified.

I believe that the amount of sodium chloride and ether used for washing in the "combined alkalinity" and "total alkalinity" determinations is quite excessive. To compare washing efficiencies, I determined the number of washings necessary to produce a neutral extract, using different volumes of the washing medium. I find from these experiments that *four* washes of 100 cc. each with a 25% solution of sodium chloride are equivalent to *five* washings of 50 cc. each or *seven* washings of 25 cc. each. Also that *four* washes of 75 cc. each with ether are equivalent to *five* washes of 40 cc. each.

With these tests in mind, I feel that it would be well to substitute *five* 50 cc. washings with salt solution in place of *four* 100 cc. washings, and *five* 40 cc. washings with ether in place of *four* 75 cc. washings where these procedures are used. Three 100 cc. washings with salt solution in most instances were not sufficient to produce a neutral extract.

I. SILVERMAN: We believe that the Solvent Method would be the most advisable for a standard method since it should give total inorganic salts, which is really what is desired.

W. S. WILLIAMS: Carbon tetrachloride was substituted in place of ethyl ether according to Mr. Pingree's suggestion. It dissolved the oil much better and troublesome cloudy precipitations were avoided.

Summary and Recommendations

The committee finds that the new solvent method for inorganic sulfates and chlorides in sulfonated oils yields concordant results, which furthermore compare favorably with the results obtained either by the Wizöff or the barium sulfate method, or by the A. L. C. A. or ash method. The new method offers the following advantages over the Wizöff method; it is simpler, more positive, and determines, besides the sulfate ion, also chlorides as well as the metals with which the sulfate and chlorine ions may be combined in the original oil. It is likewise preferable to the A. L. C. A. method because ammonium salts do not interfere with the procedure, and because it is a direct method and does not depend upon a number of other determinations, such as organically combined SO_3, fixed alkalinity, etc. for the result.

The results by the new titration methods for sodium acetate and/or sodium carbonate indicate that the meth-

ods are reliable. The manipulations are not difficult and consist mainly in the extraction of the oil by the methods developed in connection with the determination of organically combined sulfuric anhydride in the proposed A. A. T. C. C. ash-gravimetric method. It is to be noted that the new titration methods for titratable salts also yield the alkalinity corresponding to the soap in the sample.

In view of these findings the committee recommends the solvent method as a standard method for the determination of total inorganic sulfates and chlorides in sulfonated oils; it further recommends that all other salts shall be included with the "undetermined." However, if it is desired to estimate the acetates and carbonates separately, the optional titration methods may be used.

PROPOSED STANDARD METHOD OF TEST FOR INORGANIC SULFATES AND CHLORIDES IN SULFONATED OILS

A. A. T. C. C. Method

Scope:

1. This method of test determines the inorganic sulfates, chlorides, and all other salts that are insoluble in a mixture of oleic acid and carbon tetrachloride, existing in a sample of sulfonated oil by dehydrating the sample, dissolving in a solvent, filtering, and weighing the residue.

Apparatus

General:

2. Where a Gooch crucible is used, it shall be ignited in a larger crucible in which the Gooch crucible is supported by an asbestos ring.

Means of Igniting Gooch Crucible:

3. The Gooch crucible shall be ignited in a larger crucible assembled as in Fig. 1.

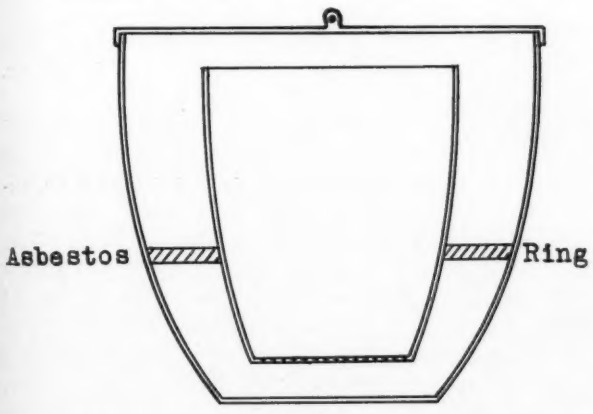

Figure 1

Filter Paper:

4. 9 cm. Whatman No. 40, or similar grade, ashless filter paper shall be used.

Reagents

Oleic Acid:

5. Oleic acid shall be the U.S.P. grade.

Carbon Tetrachloride:

6. Carbon tetrachloride U.S.P. shall be used as the solvent.

Ether:

7. Ethyl ether shall be U.S.P. grade.

8. Asbestos fiber shall be acid and alkali washed.

Procedure

General:

9. The procedure shall depend upon whether or not the sample contains ammonium salts.

In the Absence of Ammonium Salts:

10. Three to five grams of the sample shall be weighed into a 250 c.c. glass beaker, an approximately equal amount of oleic acid added, and the mixture heated in an oil bath, with constant stirring with a thermometer, at a temperature of 105° to 110° C. until practically water-free. The temperature shall now be raised to 118°-120° C. and kept there for about five minutes. The dehydrated sample upon cooling shall remain liquid otherwise more oleic acid shall be added. The dehydrated sample shall be dissolved in 100 c.c. of carbon tetrachloride, warmed to 50°-55° C., and filtered through a counterpoised filter paper or a Gooch crucible.

The Gooch crucible shall be prepared by passing through the crucible a thin emulsion of asbestos fiber in water until a pad of about 2 mms. in thickness is formed. The asbestos pad shall then be washed with water until no fibers run through. The crucible shall then be dried and ignited for 30 minutes, at first gently and finally more strongly. 75 c.c. of carbon tetrachloride shall now be passed through the crucible, again ignited, cooled in a desiccator and weighed. The process of washing with carbon tetrachloride shall be repeated until there is no further loss in weight.

The residue shall be washed with three 15-c.c. portions of a 2% solution of oleic acid in carbon tetrachloride, then with six 15-c.c. portions of hot carbon tetrachloride and finally with two 15-c.c. portions of ether or until the residue is free from oil. Care shall be taken that the top of the filter is thoroughly washed. The last traces of the residue may be transferred to the filter by allowing the solvent to evaporate when the salts become free flowing. The residue shall be dried at 125° to 130° C. for 45 minutes, cooled in a desiccator, and weighed. The heating shall be repeated until constant weight is reached. The residue shall then be ignited for 15 minutes, weighed, and the ignition repeated until the weight is constant.

In the Presence of Ammonium Salts:

11. The procedure shall be identical with the procedure in the "Absence of Ammonium Salts" with the following exceptions: (a) In preparing the Gooch crucible, it shall not be ignited but heated at 125°-130° C. for 45 minutes and the heating repeated until constant weight is reached, and (b) the residue, whether in a Gooch crucible or on filter paper, shall not be ignited but heated as in (a).

Calculations

Method:

12. The method of calculation shall depend upon whether or not ammonium salts are present in the sample.

Nonvolatile Inorganic Sulfates and Chlorides (In the Absence of Ammonium Salts):

13. The weight of the "dried" residue divided by the weight of the sample multiplied by 100 shall be the per cent of the "dried" residue. The weight of the "ignited" residue divided by the weight of the sample multiplied by 100 shall be the per cent of the "ignited" residue. The difference between the per cent "dried" residue and the per cent "ignited" residue (in the absence of ammonium salts) shall be not greater than 0.25%.

The per cent nonvolatile inorganic sulfate and chlorides shall be the per cent of the "ignited" residue, or

$$\text{Per cent nonvolatile, inorganic sulfates and chlorides} = \frac{\text{W't "ignited" residue} \times 100}{\text{W't Sample}}$$

and shall be reported as ". . . % nonvolatile, inorganic sulfates and chlorides by weight, A.A.T.T.C. Ignition Method."

Inorganic Sulfates and Chlorides, Including Volatile Salts:

14. The per cent inorganic sulfates and chlorides, including volatile salts, shall be the weight of the "dried" residue divided by the weight of the sample multiplied by 100, or

$$\text{Per cent inorganic sulfates and chlorides, including volatile salts} = \frac{\text{W't "dried" residue} \times 100}{\text{W't Sample}}$$

and shall be reported as ". . . % inorganic sulfates and chlorides, including volatile salts, by weight, A.A.T.C.C. Non-Ignition Method."

Accuracy

Accuracy:

13. Tests by various analysts indicate that the average variation from the mean result, for sulfonated oils containing a normal amount of inorganic salts, may be expected to be about 0.1%.

PROPOSED OPTIONAL METHOD OF TEST FOR ALKALI ACETATES AND CARBONATES IN SULFONATED OIL

A. A. T. C. C. Titration Methods

These methods determine the carbonates and/or acetates present in a sulfonated oil by titration. The methods, however, do not yield correct results in the presence of other titratable salts, but salts which react neutral to methyl-orange do not interfere. The titration method is varied somewhat, depending upon whether or not the sample contains carbonates, acetates, or both.

Procedure in Presence of Carbonates Only

Alkalinity as Soap (A_s). Dissolve 5 grams of the sample in 100 c.c. of water contained in a separatory funnel and add 25 grams of sodium chloride, 5 drops of 0.1% solution of methyl orange and 60 c.c. of ether. Titrate with 0.5N sulfuric acid until distinctly acid (about 2 c.c. excess), shake well, and allow the layers to separate. Wash the ether layer with two 25-c.c. portions of saturated salt solution and the combined water layers with 30 c.c. of ether. Retain the water layers for the following determination. Combine the ether layers, add exactly 0.5 c.c. of 0.5N caustic soda, evaporate the bulk of the ether, add 100 c.c. of neutral alcohol, and titrate with 0.5N sodium hydroxide until neutral to phenolpthalein. The result (as well as all other alkalinities) is expressed in mgms. KOH per gram of sample and, since there can be no free fatty acids in the presence of carbonates, represents the alkalinity corresponding to the soap (A_s).

Total Alkalinity (A_t). This test is similar to the determination of total alkali in soaps. The combined water layers from the previous analysis are boiled to expel carbon dioxide and the titration is completed. The result equals the sum of the alkalinities corresponding to the soap (A_s) and carbonate (A_c) or $A_t = A_s + A_c$. Hence, % sodium carbonate $= \frac{1}{(10)} \frac{Na_2CO_3}{2\,KOH} (A_t - A_s) = 0.09447\,(A_t - A_s)$.

Procedure in Presence of Acetates Only

Alkalinity as Free Fatty Acids (A_f). Follow the usual procedure for free fatty acids—namely, dissolve 5 to 10 grams of the sample in 100 c.c. of neutral alcohol, warm if necessary, and titrate with 0.5N sodium hydroxide until neutral to phenolphthalein. Calculate the results as mgms. KOH per gram of sample.

Combined Alkalinity as Soap (A_s) *and as Free Fatty Acids* (A_f). Dissolve 5 grams of the sample in 100 c.c. of water contained in a separatory funnel and add 25 grams of sodium chloride, 5 drops of 0.1% methyl orange solution, and 60 c.c. of ether. Add an excess of approximately 0.5N sulfuric acid (about 2 c.c. excess), shake well and allow to separate. Wash the ether layer with five 50-c.c. portions of concentrated salt solution or until neutral to phenolphthalein. Discard the water layers. The ether layer is then treated and titrated as under "alkalinity as soap" above. The result in this case represents the combined alkalinities of the soap (A_s) and of the free fatty aicds (A_f). The alkalinity of the soap is evidently the difference between this determination and the alkalinity or free fatty acids.

Total Alkalinity (A_t). Heat with constant stirring 5 grams of the sample with approximately twice its weight of oleic acid in a 250 c.c. beaker, immersed in an oil bath, for 30 minutes at 123° to 125° C.

Dissolve in 50 c.c. of water, add 5 drops of methyl orange solution and titrate with 0.5N sulfuric acid until distinctly acid (about 2 c.c. excess). Transfer to a separatory funnel, add sodium chloride to make a 25% solution and extract with five 40-c.c. portions of ether or until the ether layer is practically neutral to phenolphthalein. Discard the ether layers, rinse the funnel with water, and complete the titration in the combined water layers. The titration represents the combined alkalinities of the soap and the acetate (A_h), or $A_t = A_s + A_h$. Hence, sodium acetate may be calculated from the following formula: % sodium acetate $= \frac{(1)}{(10)} \frac{NaA_c}{KOH} (A_t - A_s) = 0.1462 (A_t - A_s)$.

Procedure in Presence of Sodium Carbonate and Sodium Acetate

Alkalinity as Soap (A_s). The fat is extracted, washed, and titrated as in "combined alkalinity as soap and as free fatty acids" above. The wash waters, however, are retained for the following test. Evidently in the presence of carbonates there can be no free fatty acids present; hence, the result represents the alkalinity corresponding to total soap (A_s).

Alkalinity as Soap (A_s) *and as Carbonate* (A_c). The combined water layers from the previous determination are heated under an air condenser to expel carbon dioxide. The condenser is then washed down with water and the titration of the contents completed until neutral to phenolphthalein. The titration obviously represents the combined alkalinities of the soap and carbonate. Hence, the difference between the last two determinations represents the alkalinity of the carbonate (A_c), from which the per cent of sodium carbonate may be calculated as follows: % sodium carbonate $= \frac{(1)}{(10)} \frac{Na_2CO_3}{KOH} A_c = 0.09447\ A_c$. ($A_h$).

Total Alkalinity (A_t). This is determined exactly as in the presence of acetates only. The result represents the combined alkalinities of soap, carbonate and acetate, or $A_t = (A_s + A_c) + A_h$. The alkalinity of the acetate may now be determined since all the other terms are known. The per cent of sodium acetate may then be calculated from the corresponding alkalinity by the following formula:

$$\% \text{ sodium acetate} = \frac{(1)}{(10)} \frac{NaA_c}{2\,KOH} (A_h) = 0.1462$$

Acknowledgment

The chairman is very grateful to the members of the subcommittee on sulfonated oils for their efforts in this work, and particularly to Mr. R. A. Pingree for the many valuable suggestions and extra experiments that he performed. This investigation is being carried out under the direction of Professor L. A. Olney, Chairman of the Research Committee of the Association.

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SOUTHEASTERN SECTION SPRING MEETING

ON Saturday, April 21, at 6:30 P.M., at the Ralston Hotel, Columbus, Georgia, the spring meeting of the Southeastern Section was held with an attendance of fifty-one. After dinner the meeting was turned over to the entertainment committee, who presented several delightful numbers. Following these was a moving picture illustrating the sanforizing process, which was furnished by Cluett, Peabody and Company.

Mr. Paul Seydel, Seydel Woolley Company, Atlanta, Georgia, then gave a paper on "The Chemical Composition of the Cotton Fiber and the Use of Metallic Chlorides; the Possible Effect These May Have on the Tensile Strength of the Material." After this most interesting and instructive paper, the discussion was led by Mr. L. L. Bamberger, of Eagle and Phenix Mills.

"Immunized Cotton" was the topic of the excellent paper read by Mr. Harvey W. Bell of the Hurlbut-Bell Company, Chattanooga, Tennessee, representing the Munitex Corporation of Paterson, New Jersey. The discussion which followed this paper was led by Mr. W. F. Christman.

Before adjournment the chairman called to our attention the need for increased enrollment, and asked those present to make an effort to secure additional members, both active and junior, throughout the section, noting that the necessary application blanks were on hand for this purpose. Since there was no further business, the meeting adjourned.

Respectfully submitted,
C. A. JONES, *Secretary*.

APPLICATIONS FOR MEMBERSHIP

Active

Marion E. Griffith—Instructor in Textiles and Research Asst., Ohio State University. Sponsors: E. E. Fickett, L. A. Olney.

Clifford Jones—Salesman, John Campbell & Co., Chicago, Ill. Sponsors: G. C. Lommell, L. I. Fidell.

William R. Howes—Textile Colorist, Calco Chem. Co., Bound Brook, N. J. Sponsors: R. R. Sleeper, G. A. Moran.

Frank Korman—33 Keeney Court, So. Manchester, Conn. Sponsors: E. C. Bertolet, H. A. Walter.

Charles E. Lennox—Chemist, Swift & Co., Chicago, Ill. Sponsors: A. T. Brainerd, G. H. Johnson.

Alexander C. Mackey—Supt. of Dyeing, Peerless Hosiery Dyeing Co., Pleasantville, N. J. Sponsors: E. C. Knaeble, A. E. Jones.

J. Ernest Meili—Chief Chemist, United Piece Dye Works, Lodi, N. J. Sponsors: D. H. Powers, R. Boehringer.

Clarence E. Moss—Salesman, John Campbell & Co., Uxbridge, Mass. Sponsors: H. J. Daigneault, Frederick Mason.

Robert Schmeidler—Chemist, United Piece Dye Works, Paterson, N. J. Sponsors: Andre Schoen, R. Boehringer.

Paul B. Seydel—Pres. & Chief Chemist, Seydel Woolley Co., Atlanta, Ga. Sponsors: C. A. Jones, C. B. Ordway.

Junior

Talbot Barnhill—Asst. Dyer, Avondale Mills, Sylacauga, Ala. Sponsors: T. G. Mungall, W. F. Crayton.

Wm. Frank Uhlig—Chemist, Calco Chem. Co., Bound Brook, N. J. Sponsors: R. R. Sleeper, D. J. Graziano.

Student

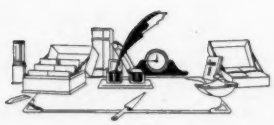
Harold Smith—Lowell Textile Institute. Sponsor: L. A. Olney.

G. Robert Thompson—Lowell Textile Institute. Sponsor: L. A. Olney.

William J. Wynn, Jr.—Lowell Textile Institute. Sponsor: L. A. Olney.

L. R. Ellenburg—Alabama Polytechnic Inst. Sponsor: C. B. Ordway.

E. E. Flowers—Alabama Polytechnic Inst. Sponsor: C. B. Ordway.



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SIMPLIFICATION

AN APPARENTLY mounting tide of criticism is being directed against so-called price fixing provisions in NRA codes. Not all of it comes from "radicals" and "parlor pinks." Robert Amory, president of the Nashua Manufacturing Co., a cotton goods manufacturer of first rank, declared in Boston on Wednesday:

"Many codes at present contain a prohibition against selling below cost. In my mind this is fundamentally wrong, in that costs are variable things and not steady, nor will they ever be subject to exact determination. These parts of codes are, in my opinion, unenforceable, and will be subject to much evasion."

On the same day in New York, Dr. Paul H. Nystrom, a noted authority on marketing, made this significant statement: "It is unthinkable that society will permit any group or industry to determine prices and market control itself at the expense of the public without public supervision."

The National Recovery Administration has approved a provision in many codes prohibiting sales below cost. It is apparently committed to approval of the inclusion in codes of a provision allowing code authorities, subject to Administration check, to set up emergency minimum cost prices below which the sale of goods is prohibited. The code authorities for both the hosiery and the knitted outerwear industries have submitted clauses of this nature as amendments to their codes, and there is every reason to believe that such clauses will receive Administration approval.

The code authorities of both these industries are composed of men who are truly representative of the best intelligence of the industries. They are following their best judgment as to what is desirable for their industries, thus disagreeing with those who are critical of price clauses in codes. Their judgment is to be respected. The difference of opinion is perhaps characteristic of the highly unusual experimental days through which we are going and gives us an idea of the complicity of the NRA structure.

In the whole recovery program there are many evidences of diametrically opposed policies, conflicting ideas as to what are the best means to a desired end, or as to the end itself. They have naturally led to confusion in the minds of the lay business man about the whole program, and must be straightened out if the program is to be more successful than it has been to date.

It seems obvious that the NRA will be rather thoroughly reorganized before many months have elapsed, and that the reorganization may consist largely of simplification. The argument is advanced by sound business men that the NRA movement has become over-extended and over-complicated, and that it probably would be desirable procedure to eliminate most of its superstructure, leaving as a basis for future building a relatively few fundamental and *enforceable* provisions covering maximum working hours, minimum wages and simple trade practice provisions designed to compel honesty in industry.

That there is a movement on foot to simplify the functioning of the NRA is shown by the recent non-acceptance of codes for many of the smaller industries which are in reality minor parts of other larger and more important industries. Many of the codes of which we have received copies in the past seem to us to have been for industries which are so small that it appears hardly neecssary to have separate codes for them because of their relative unimportance. That this view is also held by the Administrator is shown by his recent action in grouping these smaller industries under the codes of the industries of which they are a part or else have them work under the general principles of the recovery agreement. There is no doubt but what the NRA has grown much too complicated for the average person to understand and when such a point has been reached the average person loses interest and cancels his wholehearted support.

When the time comes to determine just what portions of the NRA principles will be maintained (as its promoters desire) as a permanent business "constitution" we believe that we will find that its aims and ideals will be much more simply stated and applied. It is only by doing this that industry will be able to rule itself intelligently and with as small an amount of argument and trouble as possible under the codes.

● 1934 FALL WOOLEN COLORS

New interpretations of brown are strongly emphasized in the collection of forty-six fall woolen colors just issued by the Textile Color Card Association to its members in advance swatch form.

In this outstanding basic color group, rich warm browns, rather than the colder versions, strike the newer fashion note, it was pointed out. Important variations of this theme include Malacca Brown, a new chestnut type differing from the old version, and Tabac, a brown of tobacco cast. Slightly reddish browns of mahogany and plum undertone likewise have high style prestige, as portrayed in Rose Mahogany and Grapebrown. Lively light browns of cedar cast, like Oloroso and Spanish Sherry, represent another new tendency.

Greyed and neutral browns retain a place in the fall mode, as expressed in Ascot Brown, Jaffa Brown and Cloister Brown. Cocowood and Leafdust are restrained lighter renditions of the cocoa family.

New beiges, especially chosen to be used with the fall browns, comprise Winebeige, complementary to Rose Mahogany, Candy Beige keying with Malacca Brown and other related tones, Shadobeige, a bisque shade that blends smartly with Grapebrown, and Butterbeige of yellowish cast, for accent with Tabac.

Greens give evidence of proving close rivals to browns in fashion importance, with the smart bluish and almond types of greatest style significance.

New interpretations of rust and coppery tones show every indication of repeating their popularity of last season.

Blues will be carried over into the early fall, though their volume will of course be limited.

Gay, high-spirited hues will have considerable merchandising value as smart dress shades for the cocktail or "dansant" hour, as well as for sports and casual townwear. Included in this lively color group are Leafrose, a new autumn rose; Antique Blue, a subtle greenish blue; Aztecan, a pottery rust of rosy cast; Leafgold, a mellowed old gold; Chinese Coral, a lively reddish coral; Ming Gold, a sparkling yellow gold; Manchu Green, a bright imperial green; Gloria Red, a rich rose red; Regatta Blue, a gay nautical shade and Pencil Red, a vivacious yellowish tone.

● SUMMER TEXTILE COURSES

Textile Microscopy, Textile Technical Analysis, and Textile Laboratory are the special summer courses being offered again this year by the Massachusetts Institute of Technology under the direction of Professor E. R. Schwarz. The work begins on July 25 and continues for six weeks ending on September 5.

Textile Microscopy consists of 30 lectures and 45 hours of laboratory. The lectures will be given at 10:15 each week day except Saturday and the laboratory will be offered from 1:00 to 3:30 on Monday, Wednesday, and Friday afternoons.

Textile Technical Analysis consists of 30 lectures given on each week day morning with the exception of Saturday from 9:15 to 10:15 and will cover the technique of the physical testing of textiles (exclusive of optical methods) and will also include discussion of methods of report writing, precision of measurements, graphical interpretation of data and the elements of fabric and yarn structure.

Textile Laboratory may be devoted to work in Textile Technical Analysis, to work in Textile Microscopy or both and is offered to fill a 15 hour per week schedule, arranged to suit the convenience of the student. Registration for the laboratory work is strictly limited and an early application is necessary to ensure membership in the group.

Excellent living facilities are available at very moderate cost in the new dormitory group. Reservations may be made at any time.

Groups aggregating 58 men and women from the industry and its allied branches have taken these courses during the regular school year and the opportunity afforded during the summer has always met with gratifying response in the past. At this particular time the sort of information obtainable would seem of particular value to the industry.

● ITALIAN DYESTUFFS INDUSTRY

A national dyestuffs committee has been formed in Italy for the purpose of studying the domestic dyestuffs industry and proposing measures for its protection and development, according to C. C. Concannon, Chief of the Commerce Department's Chemical Division.

Details of the Committee's exact procedure, Concannon states, are not yet available, but it is reported that a central sales bureau will be established to handle all transactions in dyes, both domestic and imported, and that licenses will be adopted for imports of dyestuffs.

Italy's domestic production of synthetic dyestuffs amounted to 6,200 metric tons in 1933 compared with 5,700 tons in 1932 and 7,500 tons in 1929, the peak year. Increased production in 1933 was accompanied by a 10 per cent rise in consumption. Imports were practically offset by exports and the volume of domestic production accounted for approximately 83 per cent of consumption. On a value basis, however, production accounted for only 50 per cent of consumption, as Italian imports consist of the finer grades of dyes. The bulk of Italy's imports of dyestuffs come from Germany and Switzerland, with small quantities originating in France and elsewhere.

United States export and import trade in dyestuffs with Italy, it is pointed out, is of insignificant proportions.

CHEMISTRY OF SILK

(Continued from page 284)

The following table was prepared from data published by Mosher in 1931.

Optimum Conditions for Enzyme Action

	pH	*Temperature*	*Time*
Pepsin	1.8	40° C.	4 hours
Trypsin	8.1 to 9.9	40° C.	8 hours
Activated Papain	5.1	80° C.	1 to 2 hours
Proteases and Yeasts	8 to 9	55° C.	4 hours

We are also indebted to Mosher for the following summary of the status of enzymes in connection with the degumming of silk:

1. In no case do enzymes effect complete removal of the sericin.

2. Increased removal of sericin is obtained by a preliminary treatment in hot acid, neutral or alkaline liquors.

3. Natural fats and coloring matter are not touched by enzymes.

4. A mild soaping after the enzyme treatment will complete the removal of the sericin, as well as the natural fats and the coloring matter.

5. Enzyme degummed silk does not show any improvement over soap degummed silk in luster, tensile strength or absence from fuzziness.

Recent patents have stressed certain improvements in the action of some of the enzymes, notably papain and the other proteases. It is possible that further studies will develop these agents to the point where they can be profitably used for certain degumming purposes.

(To be continued)

● SURVEY OF SIZING

An automatic recording viscometer is a vital need of that large part of the textile industry that uses starch pastes in the sizing of its warps or finished goods. This was one of the findings of the conference on the sizing of cotton and rayon warps held at the Hotel Commodore recently by the United States Institute for Textile Research. The effectiveness of starch pastes used in sizing depends largely upon the maintenance of a desired viscosity, but starch pastes are notoriously unstable in viscosity; therefore, the need of an effective instrument to record the change in viscosity. Even more important is the need of some salt or other chemical that will increase the stability of starch pastes; this buffering agent must be relatively cheap, since the sizing process, though necessary, is one of extremely low cost per pound of yarn or fabric.

This conference was designed by U. S. Institute for Textile Research for a scientific check-up of existing trade practice in sizing methods and materials; it being recognized that the technique of sizing is the result of empirical development, rather than of scientific research. The existing technique is similar to that in certain other equally old industries where the "how" of processing is well standardized, but with little scientific knowledge of "why" certain methods and materials are used. U. S. Institute proposes to organize a research program, based upon the findings of this conference, the results of which it is hoped will show whether or not sizing methods are scientifically correct, and whether or not they may be improved and cheapened along scientifically correct lines.

An attendance of more than 75 manufacturers and members of allied trades most of whom took part in the open discussion at morning and afternoon sessions, reflected keen interest in the subject. Dr. H. DeWitt Smith, vice-chairman of U. S. Institute's research committee, was chairman of the conference. The keynote speakers were the following: B. L. Hathorne, sizing specialist of New York City, who spoke on the technical approach to the warp sizing problem; E. R. Schwarz, professor of textile technology, Massachusetts Institute of Technology, on the relation of fiber properties and moisture regain to warp sizing; Albert Palmer, research director, Crompton & Knowles Loom Works, Worcester, Mass., on the warp sizing problem at its source—the loom. Among others who took part in the discussion were W. A. Nivling, sizing consultant, Boston, Mass.; Dr. Robert E. Rose, president of the American Association of Textile Chemists and Colorists and director, technical laboratory, E. I. du Pont de Nemours & Co., Wilmington, Del.; Howard Clayton, bleachery superintendent, Cluett, Peabody & Co., Troy, N. Y.; Prof. Louis A. Olney, Lowell (Mass.) Textile Institute; William H. Cady, chief chemist, U. S. Finishing Co., Providence, R. I.; H. M. Chase, superindent of dyeing, Riverside & Dan River Cotton Mills, Danville, Va.; Dr. Irving J. Saxl, research director, Waypoyset Mfg. Co., Central Falls, R. I.

● FABRIC TESTING DEVICES

The American Instrument Company, of Washington, D. C. has just released a new bulletin (No. 2010) which describes two instruments developed by the U. S. Bureau of Standards for the testing of fabrics and which are now available to the trade for the first time. These two instruments are the *Compressometer* which is a portable instrument for measuring thickness, compressibility and resilience of woven, knitted and pile fabrics and the *Fabric Air Permeometer* which measures the extent to which a fabric is penetrated by air at a low pressure. Copies of this bulletin may be obtained by writing to the above mentioned company at 774 Girard St., N. W., in Washington.

OPEN FORUM

Rules under which this department of the REPORTER is conducted are as follows:

1. Anyone who has a technical problem on which he would like advice may submit it to the Reporter and it will be published in the first available issue.

2. Any reader who feels that he has a solution of the problem may forward his reply to us and if we feel that the reply is suitable it will be published in a succeeding issue.

3. For each such reply published the Reporter will pay a minimum of $2.

4. In case there is more than one answer to the same question, the one covering the subject in the most complete and authentic manner will be published, or if two answers are sufficiently different both will be published. If two answers are considered equally good, the one first received will be given priority.

5. The Reporter will assume no responsibility whatsoever for the accuracy of the answers submitted—it is simply offering a vehicle for the public expression of its readers' opinions.

6. Both the questioner and answerer must send in his complete name and address. When requested we will publish only the initials or other identifying insignia.

ANSWERS

4—Potassium permanganate has at all times been used in small amounts by the textile industry for certain processes of bleaching and dyeing. Various manufacturers of this product would like to know whether or not its limited used in various textile processes is due mostly to its cost, or because other bleaching agents are more suitable. Also, if its use could be extended beyond the process of bleaching if its cost was reduced somewhat.—**Chemical Manufacturer.**

Answer No. 1—Potassium permangenate finds very limited application in the textile trade for the following reasons: As a bleaching agent, its action is usually too drastic and without great care in working, tendering will easily result. Also, ensuring a perfect solution of potassium permangenate is very difficult in practise. Further, it is a very tedious job clearing afterwards with oxalic acid or sodium bisulphite, the latter being particularly unpleasant. I do not think the price factor is an important consideration.

I consider it most unlikely that its use in the textile trade would be extended, were a reduction in price possible. Apart from mineral dyeing which is rapidly becoming obsolete, it is mostly used for such small jobs as stripping overdyed pieces, clearing aniline black prints and the like. It is sometimes used in linen bleaching. In all these latter cases, the quantities used are so small, that I do not think the finisher would fall out over the price, even in these difficult times.—A. I. C.

Answer No. 2—The use of the permangenates in belaching is mentioned in almost every textbook but like the weather, nobody does anything about it.

Quite probably it was the world war which, by shutting off the supply of potassium permangenate, drove textile bleachers to the use of peroxide as an alternative bleaching agent.

I have used permangenate as a bleach—most everybody has—and it is quite successful in producing a half bleach but I have never been able using it to produce a brilliant full bleach. This most likely is due to my not knowing how to handle the permangenate properly.

There is, in my belief, a distinct field for the use of the permanganates as bleaching agents particularly in the unexplored field of continuous bleaching.

Permanganates should be an ideal bleaching agent and all that we need is for the manufacturers of the product to get together and establish some competent chemist and bleacher with funds for a year or so to work out the necessary close control.

The introduction of the Igepons, Gardinols and similar compounds, being wetting agents that are not precipitated by acids, opened the way to successful use of permanganates as bleaching agents.

I intend to take up again their use and I feel sure that a little intelligent promotionary work by the makers of the permanganates will establish a market. —H. S.

5.—We are interested in obtaining some information on the reaction of rayon to dyestuffs in comparison to silk, cotton, wool and linen. Can you set us straight on the following points?

A. Does rayon take dyes more easily than any, or all, of these?

B. Does rayon take truer colors of dye?

C. Does it take dyes more evenly?

D. Does it retain colors longer?

E. Can a greater range of color variation be obtained in dyeing rayon? More subtle colors? More brilliant?—**M. J.**

Answer: The animal fibers, silk and wool, are so diametrically opposite to cotton, linen and rayon (viscose) that no comparison can be taken, and the question raised is somewhat absurd.

The following table shows the affinities of the different fibers towards different classes of dyestuffs.

B.—In the writer's opinion, the question cannot be discussed, as no definite shade can be laid down and it

Dyestuff	*Wool* *Silk*	*Rayon* *Cotton* *Linen*
Substantive	Most dye wool and silk equally in neutral or acid bath. Benzo Red 10B, Chloramine Yellow and a few others act more powerfully on silk. Geramine G, Benzo Blue 2B and a few others act more on wool.	All have good and practically equal affinity for the three fibers.
Basic	Most basic dyestuffs act more powerfully on silk than wool. Saffranine and methylene blue when dyed in cold acetic bath dye silk preferentially.	Little direct affinity, but may be satisfactorily applied on suitable mordant. Viscose may be mordanted before dyeing.
Acid	Most acid dyestuffs dye wool and silk to same extent. A whole series, acid fuchsine, the eosines, etc. have less affinity for silk.	Practically without exception, these dyestuffs are inapplicable to these fibers.
Vat	Silk may be satisfactorily dyed by general process, but on account of caustic, wool is not generally dyed by the same method.	As a general rule, there is little difference on the three fibers. Viscose often appears darker than cotton with mixtures. (This varies with manufacture of silk.)
Mordant	Employed only to a restricted extent on silk, on account of bad effect of mordant. Extensively used on wool.	Apparently equal affinity.

said: "That is the true shade of the dyestuff, no matter what fiber it be dyed upon." For example, Caledon Golden Orange 3GS invariably appears very slightly redder on viscose than cotton, and one can only conclude that each represents the true color on each particular fiber.

C.—As a general rule, I think it is fairly safe to state that rayon dyeings do appear more level dyed than similar dyeings on other fibers, but this is probably due to the fine nature of the filament and is actually no criterion when it comes to the question of equal distribution of the dye particles.

D.—The writer does not know of many observations that have been made with regard to the respective fastnesses compared to rayon, but the fastness to light of vat dyestuffs on rayon is slightly different to that of cotton.

E.—Yes, more brilliant and subtle shades can be obtained on rayon than most other fibers, due presumably to the natural brightness and brilliancy of the fiber itself. **—A. I. C.**

NEW QUESTIONS

6—I would appreciate it if you could give me any definite information or refer me to a source in regard to any present developments in the use of spun glass for decorative purposes on woolens and worsteds.—**M. M.**

7—I would like a preparation or mordant which, when applied to certain parts of woolen carpet yarn (in skeins), would resist dyeing in an acid dye bath.—**W. K. S.**

8—Please give me a method for stripping of all dyes from old woolen garments, mainly sweaters.—**D. W. C.**

BOOK REVIEW

Rayon and Synthetic Yarn Handbook, by E. W. K. Schwarz and Herbert R. Mauersberger, 420 pages, $4.00, Rayon Publishing Company, New York.

According to the authors' preface in this book it is designed to furnish a reference book on the technical and statistical side of the American rayon industry which is all-inclusive and written in semi-technical language. The method in which this designation is carried out may best be shown by listing the chapter contents of the book, which are as follows:

Chapter I—Historical background, Chapter II—Cellulose and the four main manufacturing processes, Chapter III—Manufacture of allied rayon yarns and materials, Chapter IV—Economic and statistical background (by Stanley B. Hunt, Editor, "The Textile Organon"), Chapter V—Physical and chemical properties, Chapter VI—Denier, Chapter VII—Conversion into woven grey goods, Chapter VIII—Commercial constructions of staple synthetic grey goods, Chapter IX—Conversion into knitgoods and hosiery, Chapter X—Dyeing, Chapter XI—Printing, Chapter XII—Dry finishing and special processes, Chapter XIII—Analysis and testing (including A.A.T.C.C. Rayon Subcommittee report of rayon identification), Chapter XIV—Trade marking, Chapter XV—Synthetic yarn producing companies of the world, Chapter XVI—Literature.

The book is complete with numerous illustrations, diagrams and tables. For the rayon student, technician, manufacturer, seller or retailer there is a good deal of valuable information gathered in this book.

● GENERAL RELEASES

The General Dyestuffs Corporation has prepared circulars on the following new releases. Copies of these cards may be obtained on request.

Indanthren Printing Black T L Suprafix—said to produce on cotton and rayon a deep jet black, and in reductions, very pleasing shades of neutral grey. The prints are said to possess excellent fastness properties.

Indanthren Olive G B Dbl. Paste—a new product which is said to possess excellent fastness to light and weather. It is recommended by the manufacturer for the dyeing of awnings and other goods which have to stand severe exposure.

Acid Brilliant Blue R Extra—a new level dyeing acid dyestuff said to produce bright blue shades of fairly good fastness to water, salt-water, perspiration, washing and fulling. It is further claimed that the fastness to hot pressing, decatizing, carbonizing and stoving, as well as to alkali and rubbing is good. The fastness to light is only moderate. It is said to have good affinity for silk and is also recommended by the manufacturer for application printing on wool or pure silk.

20% Fast Color Salt of Fast Red K B Base—on Naphtol AS-SW impregnation is said to represent one of the brightest and most popular red combinations in the Naphtol field. The salt is stable and with an addition of Diazopon A is said to produce a stable diazo solution.

● APPOINTED BRANCH MANAGER

P. C. Zell has been appointed Philadelphia Branch Manager of Nyanza Color & Chemical Company of New York. Mr. Zell has been in the dyestuff business over 30 years, of which 15 were spent in Japan with the Chemische Griesheim-Electron. He has been with the Nyanza company since 1930 acting as demonstrator in charge of the Piedmont Section.

● TUBE-TEX MACHINES

A new kind of selling cooperation was witnessed at the Knitting Arts Exhibition in Philadelphia when plant executives, who had already leased Tube-Tex machines, actually talked to their own competitors about the merits of the machines. This friendly cooperation undoubtedly was a factor in the leasing of eleven Tube-Tex machines at the Exhibition. Delivery of machines to licensees is to commence at once.

An invitation on the wall behind the Tube-Tex invited persons interested to bring their own fabrics and see them finished on the machine. Various kinds of fabrics finished on the Tube-Tex machine were exhibited through the courtesy of the Tubize Chatillon Corporation.

The Tube-Tex on exhibit was what is known as a combination "C" machine which extracts, processes, dries, conditions and folds tubular knit goods in one continuous operation. It is operated by one man and has a production of 350 to 450 pounds per hour with a speed range from 25 to 35 yards per minute. The claim made for this machine is that it saves 10 to 35% in power and 50 to 75% in space, as well as materially lowering labor costs. There are several other types of Tube-Tex ranges on which a new bulletin has just been issued by H. W. Butterworth & Sons Company, Philadelphia, makers and licensed agents for Tube-Tex machines. Patents are held by the Tubular Textile Machinery Corporation, New York City.

● GERMANY TRADES CHEMICALS

The willingness of Germany's foreign bondholders to accept cash settlements at discounts ranging up to 25% has been of great assistance to the countries' chemical exporters who have accepted them in payment at face value, according to C. C. Concannon, Chief, Chemical Division, Bureau of Foreign and Domestic Commerce.

That this fact is largely responsible for the remarkably fine showing made by Germany's chemical exporters in recent years is borne out by a report from Consul Sydney B. Redecker, Frankfort-on-Main. This report points out that despite the fact that Germany is nominally on the gold standard, it has been able to compete with countries where exchange is greatly depreciated.

Exports of chemicals from Germany in 1933 were valued at 695 million marks, a reduction of only 4½% from the preceding year, and in terms of tonnage many leading products registered notable expansion. At the close of 1933 approximately 350,000 persons were employed, representing a reduction of 20% in unemployment accredited to that industry at the beginning of the year.

Heavy price cuts in foreign quotations made by German exporters of many chemical products would not have been possible, Mr. Concannon pointed out, had not foreign bondholders been willing to take cash settlements at substantial discounts.

The supply of these frozen credits has been greatly reduced in recent months, and the discount has declined, depriving the exporter of some of his advantage. German dollar-bonds falling due during the first half of 1934, however, may possibly bring larger quantities into the market.

NEW PATENTS
Abstracted by S.O.C.M.A.

Process for Manufacturing Sulfur Dyestuffs. (An improved process which comprises adding during the process of sulfuration of a para-amino-phenol derivative an oxidizing agent of the group consisting of air and nitro compounds to the sulfuration mass comprising an indophenol compound and an alkali metal polysulfide; a continuation of application Ser. No. 328,058, filed December 22, 1928.) George Kalischer, Frankfort-on-the-Main, and Heinrich Ritter, Frankfort-on-the-Main-Fechenheim, Ger., assignors to General Aniline Wks., Inc., N. Y., N. Y., No. 1,952,029, March 20, 1934.

Azo-Dyestuff Containing Chromium and Process of Making the Same. (For instance, from diazotized 1-amino-2-hydroxynaphthalene-4-sulfonic acid and *b*-naphthol, marine blue dyestuffs are obtained dyeing uniformly even in deep tints and fast in every respect; also possible to convert chrome Fast Cyanine into cheap and fast chromium compounds which dye marine blue.) Fritz Straub and Hermann Schneider, Riehen, near Basel, Switzerland, assignors to the firm Society of Chemical Industry in Basle, Basel, Switzerland, No. 1,946,951, February 13, 1934.

Azo Dyes. (Obtained by diazotizing an amino-aryl carboxylamide derivative containing no sulfonic or carboxylic groups, coupling with an aromatic amine of the benzene or naphthalene series which can couple in the position para to the amino group and contains no sulfonic or carboxylic groups, diazotizing the resulting amino azo compound and coupling with a naphthol or naphthol derivative containing no sulfonic or carboxylic groups; especially valuable for coloring organic liquids and solids, particularly hydrocarbons, and halogenated hydrocarbons, either aliphatic or aromatic, such as, for example, gasoline, ethylene dibromide, carbon tetrachloride, paraffin and halowax (chlorinated naphthalene.) Harold Edward Woodward, Penns Grove, N. J., assignor to E. I. duPont de Nemours & Co., Wilmington, Del., No. 1,947,027, February 13, 1934.

Azo Dyes and Methods for their Preparation. (Relates particularly to insoluble monoazo dyes ranging from yellow to orange in color and valuable for use as pigments in printing inks and paints; for instance, comprises coupling diazotized 4-chlor-2-nitroaniline with aceto-acet-2-5-dimethoxy-anilide.) Harold Edward Woodward, Penns Grove, N. J., assignor to E. I. duPont de Nemours & Co. Wilmington, Del., No. 1,947,028, February 13, 1934.

Vat Dyestuffs of the Dibenzanthrone Series and Process of Preparing the Same. (The bluish-green to yellowish-green dyestuffs of the di-benzanthrone series, which are substantially identical with the dyestuffs obtainable by condensing dihydroxy-dibenzanthrone in nitro-benzene solution with the di-(p-toluene-sulfonic acid) ester of diethylene glycol under reflux conditions and in the presence of an alkali metal carbonate.) Edward T. Howell, Milwaukee, Wis., assignor, by mesne assignments, to E. I. duPont de Nemours & Co., No. 1,947,047, February 13, 1934.

Manufacture of Soluble Compositions and Their Use in Dyeing and Printing. (A formaldehyde-bisulfite compound of a primary arylamine free from azo groups, an alkali nitrite, and an arylamide of 2-3-hydroxy-naphthoic acid.) Mordecai Mendoza, West Didsbury, Arthur George Murray, Whitefield, and Herbert Bradley Briggs, South Reddish, Eng., assignors to Imperial Chemical Industries, Ltd., No. 1,947,433, February 13, 1934.

Indigoid Vat-Dyestuffs and Process of Making Same. (For example, comprises condensing the para-dimethylaminoanil of 5-chloro-6-methyl-3-hydroxythionaphthene with 2:1-naphthoxythiophene; dye cotton fast scarlet to red, viloet and grey tints.) Jakob Muller, Munchenstein, near Basel, and Robert Stocker, Basel, Switzerland, assignors to firm: Society of Chemical Industry, Basle, Basel, Switzerland, No. 1,947,655, February 20, 1934.

New Aniline-Sulfonic Acids. (Valuable intermediate products for the synthesis of various kinds of dyestuffs.) Erich Fischer, Bad Soden-on-the-Taunus, and Albert Kissling and Herbert Kracker, Frankfort-on-the-Main, Ger., assignors to General Aniline Works, Inc., N. Y., N. Y., No. 1,947,837, February 20, 1934.

Process of Preparing Condensation Products of the Benzanthrone Series and New Products Obtainable Thereby. (Mordant dyestuffs particularly suitable for dyeing wool, possessing very good properties to fulling, potting and light.) Georg Kraenzlein, Frankfort-on-the-Main-Hochst, and Martin Corell, Frankfort-on-the-Main, Ger., assignors to General Aniline Works, Inc., N. Y., N. Y., No. 1,947,943, February 20, 1934.

Azo Dyes. (More specifically comprises monodiazotizing 1-methyl-2.4-diamino-5-nitrobenzene and coupling the monodiazo compound with 8-hydroxynaphthalene disulfonic acid-1-azo-resorcinol in a alkaline medium; capable of dyeing acetate silk fast orange tints, wool orange-brown and bordeaux tints and leather brown tints; a modification of U. S. Patent No. 1,896,710, dated February 7, 1933.) Werner Lange, Dessau-Ziebigk in Anhalt, Ger., assignor to General Aniline Wks., Inc., N. Y., N. Y., No. 1,947,945, February 20, 1934.

Azodyestuffs Insoluble in Water and Process of Making Same. (Obtained by combining any diazo-, tetrazo- or diazoazo compound with a 4-(2'-hydroxy-3'-naphthoylamino)-diphenylamine compound, none of these components containing sulfonic or carboxylic acid groups; may be produced in substance and yield when mixed with the usual substrata valuable color lakes, or prepared on the fiber yielding dyeings and printings of an excellent fastness especially to light, the shades varying from claret red to black.) Leopold Laska and Arthur Zitscher, Of-

fenbach-on-the-Main, Ger., assignors to General Aniline Wks., Inc., N. Y., N. Y., No. 1,947,946, February 20, 1934.

Acidyl Polyalkylene Polyamines. (Valuable intermediate substances in the manufacture of wetting agents some of them being used as such immediately.) Wilhelm Neelmeier, Theodor Nocken, and Hermann Friedrich, Leverkusen-on-the Rhine, Ger., assignors, by mesne assignments to General Aniline Wks., Inc., N. Y., N. Y., No. 1,947,951, February 20, 1934.

Compositions and Solutions for Use in Dyeing and Textile Printing. (As a new composition of matter a dry mixture comprising a water-soluble bromate, a water-soluble bromide, and an N-sulfonic acid.) Mordecai Mendoza, West Didsbury, and Arthur George Murray, Whitefield, Eng., assignors to Imperial Chemical Industries Ltd., No. 1,948,589, February 27, 1934.

Azo-Dyestuffs. (Obtained by coupling an arylide of 2:3-hydroxy-naphthoic acid with a diazo- or poly-azo-compound; the new dyestuffs are produced on the fiber in known manner and yield Bordeaux to violet, blue, green and black dyeings of good fullness and fastness.) Gerold Bonhote and Carl Apotheker, Basel, Switzerland, assignors to Society of Chemical Industry in Basle, Basel, Switzerland, No. 949,142, February 27, 1934.

Dispersed Pigment From Organic Carbonaceous Earth and Process for Producing the Same. (A chemically dispersed dye comprising humic material, one of the common commercial concentrate dmineral acids, an alkyl ether of the ethylene glycols and benzene; usually brown in color, varying from blackish brown to reddish-brown; for use in wood stains or for coloring other materials; contains subject matter in common with Patents Nos. 1,877,748, dated September 13, 1929, and 1,898,247, dated February 21, 1933.) Leonard P. Dove, Hinsdale, Ill., No. 1,949,147, February 27, 1934.

Process of Preparing 1.4.5.8-Naphthalene-Tetra-Carboxylic Acid and Substitution Products Thereof. (Comprises treating 1.4.5.8-naphthalene-tetra-carboxylic acid diimide, derivatives or substitution products thereof with concenrtated sulfuric acid; a valuable starting material for the preparation of dyestuffs.) Heinrich Greune and Wilhelm Erkert, Frankfort-on-the-Main-Hochst, Ger., assignors to General Aniline Wks., Inc., N. Y., N. Y., No. 1,949,202, February 27, 1934.

Bromination Products of the Dibenzanthrone Series. (Dyeing the vegetable fibre blue to violet shades from a blue vat; a continuation in part of copending application Serial No. 345,880, filed March 9, 1929, now Patent No. 1,882,285.) Max Albert Kunz, Mannheim, and Karl Koerberle, Ludwigshafen-on-the-Rhine, Ger., assignors to General Aniline Wks., Inc., N. Y., N. Y., No. 1,949,209, February 27, 1934.

Azo Dyestuff. (Obtainable, for example, from the diazo compound of 1-hydroxy-2-amino-6-methylbenzene-4-sulfonic acid and 1-phenyl-5-pyrazolone-3-carboxylic acid amide; dyes wool orange shades which by after-chroming are converted into red shades of excellent fastness properties.) Hermann Winkeler and Hans Reindel, Ludwigshaften-on-the-Rhine, and Guido Freiherr von Rosenberg, Mannheim, Ger., assignors to General Aniline Wks., Inc., N. Y., N. Y., No. 1,949,228, February 27, 1934.

Phenolic Compound of Color Bases. (The organic reaction product of a basic dyestuff of the triphenyl-methane series with a pyrazolone; for use as colored pigments, lakes, etc., and for dyeing or coloring lacquers, varnishes, pyroxylin compositions, artificial and synthetic resins, enamels, and the like). Ralph B. Payne, Elma, N. Y., assignor to National Aniline and Chemical Co., Inc., N. Y., N. Y., No. 1,953,309, 4/3/34.

Treatment of Cellulose Esters. (To effect stabilization, viscosity reduction, bleaching and clarification thereof; for instance, reducing the viscosity of cellulose nitrate which comprises heating the nitrate in an undissolved state in water containing hydrogen peroxide, and an alkali.) Roderick K. Eskew, East Orange, N. J., assignor to Du Pont Viscoloid Co., Wilmington, Del., No. 1,953,398, 4/3/34.

Chlorinated Dibenzanthrones. (Vat dyestuffs giving violet, navy blue to dark blue dyeings of special fastness to water.) Max Albert Kunz, Mannheim, and Karl Koeberle and Erich Berthold, Ludwigshafen-on-the-Rhine, Ger., assignors to General Aniline Wks., Inc., N. Y., N. Y., No. 1,953,415, 4/3/34.

Disazo Dyestuffs. (Obtainable by tetrazotizing a diamino-diphenylsulfone and coupling one mol with one mol of an ortho-hydroxy-carboxylic acid of the benzene series and one mol of a 2-arylamino-8-naphthol-6-sulfonic acid, wherein the aryl radical is one of the benzene series; yield on wool brown dyeings of good fastness to washing and fulling which turn to fast yellowish-brown tints when after-chromed.) Adolf Sieglitz and Konrad Stenger, Frankfort-on-the-Main, Ger., assignors to General Aniline Wks., Inc., N. Y., N. Y., No. 1,953,512, 4/3/34.

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